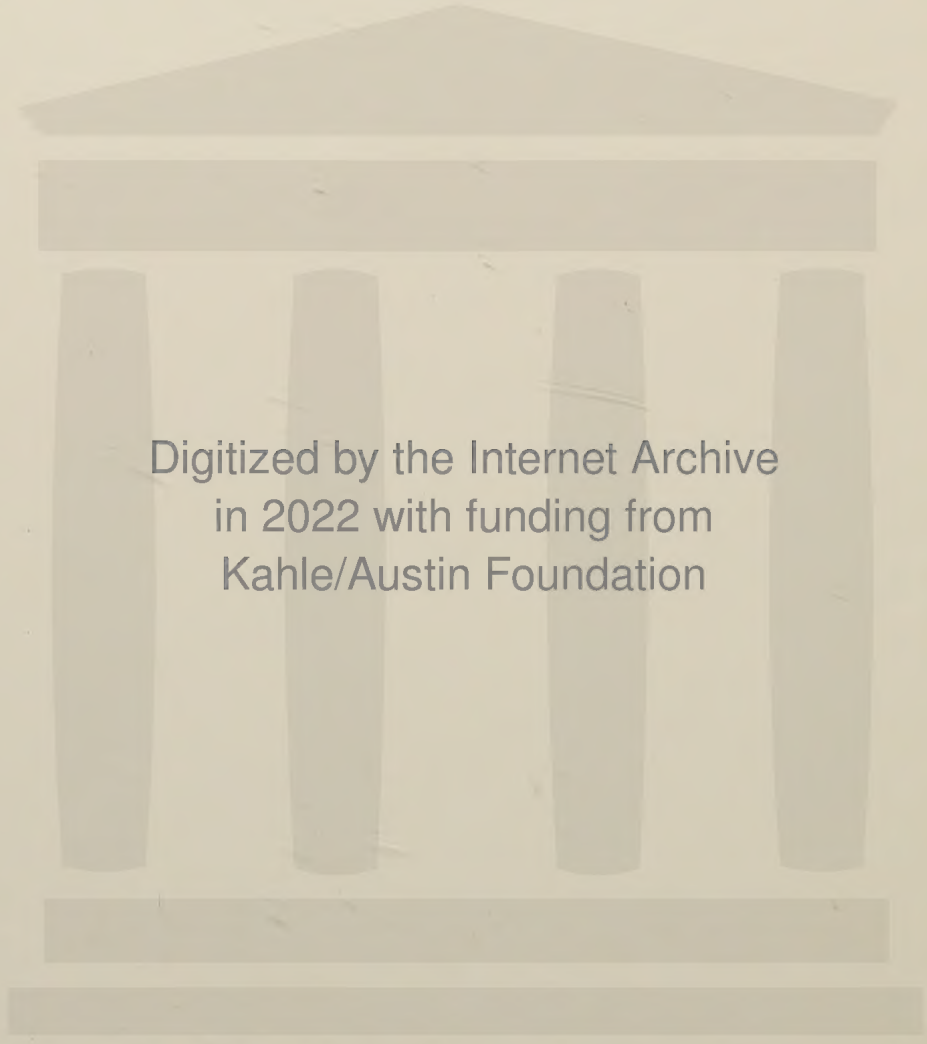
The Grapefruit

Its Composition, Physiology, and Products

WALTON B. SINCLAIR

UNIVERSITY OF CALIFORNIA
DIVISION OF AGRICULTURAL SCIENCES



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The Grapefruit

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WALTON B. SINCLAIR

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Preface

THIS VOLUME concerns grapefruit (*Citrus paradisi* MacFad.), and not pummelo (*C. grandis* Osbeck), which is grown primarily in southeastern Asia and is often referred to by the western world as the "shaddock."

For a long time after its introduction into the United States (Florida) in the early part of the nineteenth century, grapefruit was considered to be of no particular value. It was grown merely as a curiosity; during the latter part of the maturing season, the ground under the trees was covered with fruit, which was allowed to rot where it lay. Gradually, the residents began to recognize that the fruit was refreshing and wholesome, and this information was passed on to tourists from the North.

The first commercial shipments of grapefruit were from Florida to Philadelphia and New York sometime between 1880 and 1885. The first shipments are said to have netted to growers only about 50 cents a barrel, but soon the demand for grapefruit became established, and better prices were obtained. At present, it is one of our most important fruits, with an annual production averaging over 46 million boxes between 1963–64 and 1968–69. During recent years, about half of this total has been processed. Thus, from a small, discouraging beginning, the grapefruit industry has come to occupy a very important place in the economy of Florida and California and a dominant position in Texas and Arizona.

Although the United States produces about 89 per cent of all the grapefruit in the world, commercial growing of this fruit has been extended to most other citrus-producing areas—Palestine, South Africa, Puerto Rico, Cuba, Jamaica, Brazil, and many others—and has become well known in all the leading citrus markets.

McNair (1926–27) stated that Puerto Rico did not begin to produce grapefruit commercially until 1898, when it was taken over by the United States.

This volume attempts to bring together a considerable portion of the widely scattered information concerning the history, distribution, production, composition, and products, as well as the industrial, economic, and other values of the grapefruit. It is also planned to bring together the published data on the biochemical and physiological changes taking place in grapefruit during growth and maturation of the fruit on the tree. A large amount of literature has accumulated on the chemical and physiological changes of postharvest grapefruit. These data are brought together and correlated with the behavior of the fruit in storage and transit. An attempt is

made to present, as fully as the facts permit, a biochemical interpretation of all the phenomena reported. Through the application of modern precision methods and instrumentation to citrus fruits, the accumulation of biochemical and physiological data has increased at a sufficient rate to encourage an attempt, with some degree of success, at biochemical interpretation of the metabolic changes occurring in grapefruit.

Research on grapefruit has increased with the economic importance of the fruit and with its overproduction, that is, the excess amount that cannot be marketed as fresh fruit. To use these surplus supplies in commercial channels, they have to be processed into nonperishable products that can be stored for future use. To solve the enormous technical problems involved in the processing of citrus fruits, both federal and state agencies have annually contributed substantial amounts for both talent and equipment to investigate the fundamental problems involved in the whole area of food technology. The published results of these investigations have contributed significantly to our knowledge of the chemistry and physiology of the grapefruit.

The present volume is not meant to give a complete bibliography of the grapefruit, but an attempt has been made to use the pertinent data from what were considered to be the most important references, and to use these data in a logical discussion of the subject. It has been specifically designed, therefore, to present the facts systematically and to develop the data within a historical framework, together with illustrative material drawn from the accumulated literature. Enough detail is given on each topic to make the book a satisfactory source of reference in its own right, and not merely an index to the literature. In this respect, the inclusion of a large number of tables and graphs from the original papers will be particularly useful to those interested in the chemistry and technology of the citrus industry.

Because of the nature of this fruit, such information should be of value to research students, food technologists, teachers, dietitians, nutritionists, physicians, and others who are engaged in planning diets or in calculating the nutritive value of foods.

Grateful acknowledgment is made to Grant V. Wallace, of Agricultural Publications, University of California (Berkeley), for his valuable editorial contributions when preparing this book for the press. His long experience in editing scientific manuscripts and background of practical knowledge of the printing craft were the sources of his thoroughness and competence.

Finally, sincere appreciation is expressed to the many whose previously published figures and tables have been used; to those from all citrus-growing areas who furnished similar data through personal correspondence; and to all others, even though not specifically mentioned, who helped in many ways to make this publication possible.

WALTON B. SINCLAIR, December, 1971

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Chapter 1

Origin and History of the Grapefruit

THE ORIGIN of the grapefruit is not definitely known. Those who have made a careful study of the problem state that it apparently did not come from China or adjacent areas in the southeastern portion of Asia where some citrus fruits are native, or from any of the European citrus-growing areas. Grapefruit has been introduced and grown in Europe only in comparatively recent years.

Sloane (1696, 1707) described the shaddock as growing in Jamaica by 1696 and in Barbados by 1707, but he did not mention the "forbidden fruit," or grapefruit. Hughes (1750) was the first to mention it as being found in Barbados, and Browne (1789) reported it from Jamaica under the names "forbidden fruit" and "smaller shaddock." Marloth (1952), who discussed briefly the origin and early distribution of citrus fruits, said that some grapefruit trees were growing in South Africa as early as 1661.

About 1809, a Spanish nobleman, "Don Phillippe" [Felipe], migrated to Florida and settled near what is now locally known as Green Springs, in Pinellas County. He was said to have brought grapefruit seeds with him, and from them to have developed a small grove, the first grapefruit planting in the state (Hume, 1926). Just where Phillippe obtained his seeds, and the exact date of planting, were not stated. There appear to be at least 2 explanations as to the source of the seeds. A citrus fruit called the "grapefruit" was growing in Jamaica (Lunan, 1814), and this may have been the source. On the other hand, Duncan (1892), a pioneer grower in the same county, who named and introduced the Duncan grapefruit, stated that "fifty years ago Phillippe planted seeds 'of grapefruit' which came from Cuba." At any rate, he could not have obtained his seeds from Europe or Asia since, as stated above, the grapefruit was not known in those areas until much later than his time.

Phillippe gave seeds from his grove to many of his neighbors, and many of the old seedling grapefruit trees in Florida are said to have come from this source.

Tussac (1808–27) described a variety of shaddock as a fruit not larger than a good orange, which was borne in bunches like grapes and which was designated as the "forbidden fruit" or "smaller shaddock." Apparently, the variety

known to the early writers as the "forbidden fruit" was known also, at least in Jamaica, as the "grapefruit." It thus appears reasonable to assume that the name "grapefruit" originated in Jamaica, and has been used since 1814.

For a more detailed discussion of the origin and early distribution of the grapefruit, see Hume (1926, 1935), Webber and Batchelor (1943), Chandler (1950), Magness (1951), Marloth (1952), and Hodgson (1955, 1967).

The early American literature on citrus shows that all research was originally devoted to the improvement of the orange.

Relationship to Shaddock

IT SEEMS CERTAIN that the grapefruit is very closely related to the pummelo, or shaddock, and was derived from it or from a common ancestral type. The grapefruit was isolated from the pummelo as a distinct species by MacFadyen (1830, 1837), who gave it its present designation, *Citrus paradisi*.

Webber (1943) recorded in considerable detail the taxonomic relationships between the grapefruit and the pummelo. He noted the existence of many intermediate forms that link the 2 groups together, and the lack of known distinctive characteristics that will segregate all types. Some of the comparative differences between the 2 groups are listed here.

GRAPEFRUIT (*Citrus paradisi*)	PUMMELO (*Citrus grandis*)
Twigs glabrous.	Young twigs pubescent.
Leaves glabrous.	Leaves puberulent beneath, along midribs.
Petioles broadly winged.	Petioles very broadly winged.
Fruit size medium to large.	Fruit size large to very large.
Fruits mainly oblate, spherical, or obovate.	Fruits mainly spherical, obovate, or pyriform.
Rind thin to medium thick.	Rind thick to very thick.
Segments closed at center.	Segments mainly open at center.
Axis solid or semihollow.	Axis semihollow or hollow.
Pulp tender and melting.	Pulp tough and solid.
Seeds polyembryonic.	Seeds monoembryonic.
Fruits mainly born in clusters.	Fruits mainly borne singly.

Webber (1900*a*, *b*) and Frost (1926) reported that the seeds of grapefruit were polyembryonic, while those of the pummelo were monoembryonic. Webber (1943) stated that if this difference between the varieties of the 2 groups were found to be general, it would constitute one of the most fundamental distinctions between them. (See Frost, 1926.)

Varieties

THE GRAPEFRUIT GROWER, like other growers, is always on the lookout for varieties that will enable him to produce greater yields of a product that will appeal to consumers. As pointed out by Webber (1943), commercial citrus

varieties have become recognized because of special physical and chemical characteristics of the fruit. Thus, the purpose of varietal selection is to obtain representative types within the species which possess the most desirable characteristics in relation to yield, fruit size, shape, color, smoothness of rind, pulp texture, juiciness, and flavor, as well as good growth, resistance to disease, and earliness or lateness of maturity. A citrus variety is a group of plants, all having the same characteristics, that have been propagated from 1 parent individual or branch of an individual. In terms of its definition, a citrus variety (cultivar) which has descended from a single parent tree or individual mutant branch can be multiplied by vegetative or asexual methods, such as budding and grafting. Since these individuals have descended from the same ancestral source, they all should have the same genetic constitution. When variation occurs within this variety sufficiently great to change the type, the result is considered to belong to another variety because the genetic constitution has been altered. When such a bud variant is segregated and propagated, it is recognized as a new variety and, if it has practical characteristics, it may properly be developed into an important cultivar. Citrus varieties are likely to undergo changes, even when propagated only asexually.

Webber and Batchelor (1943) noted that the most important varieties of pallid-pulp grapefruit in the United States were the Marsh (Marsh Seedless) and Duncan. It is still true that the principal grapefruit grown in all parts of the world, including the United States, is the Marsh Seedless. The most prominent and best-known seedy grapefruit of commercial importance is the Duncan variety, grown chiefly in Florida for processing purposes. The Duncan is probably the oldest clone grown in Florida, for it was designated as a variety in the early 1890's by A. L. Duncan (1892). Its parentage was traced back to a seedling tree planted in 1830 (Webber, 1943). The Walters, a seedy variety, is little grown today, but has horticultural significance in that it has served as a source of buds from which 2 varieties—Foster, the first pink-fleshed variety, and Cecily, a seedless South African variety similar to Marsh—have been derived. Webber and Batchelor listed some 17 other varieties, most of which have been tested. Four of these (the McCarty, Royal, Triumph, and Cecily) are grown to a very limited extent, chiefly on an experimental basis, in local and foreign grapefruit-growing areas, but the rest are no longer propagated. Most of these varieties never attained commercial importance.

In addition to the pallid-pulp group, there are the pigmented grapefruit varieties: Foster (Foster Pink), Thompson (Marsh Pink), Ruby (Henninger's Ruby, Ruby Red, Marsh Red, Redblush), and Webb (Webb's Redblush Seedless). Much of the older acreage in Florida is planted to seedling trees; the fruit from these trees is seedy, and is known as "Florida common."

Origins. The early history of some of the older grapefruit varieties is not well known, but it seems probable that almost all the varieties now cultivated

had their origin in Florida, where the grapefruit industry began. These early varieties were seedlings that were selected and propagated because of certain superior characteristics of both fruit and tree; they are classed as Florida seedling types. The products of these varieties are so similar that it is difficult to differentiate them according to fruit characteristics. The Marsh White, with no pink coloration in its fruit, is well authenticated as having arisen as a seedling or a root sprout from a seedy white grapefruit in Florida (Webber, 1943; Waibel, 1953). It is the most common, nearly seedless, commercial grapefruit in the United States and in most other grapefruit-producing areas.

The Foster (Foster Pink) originated in Florida as a limb sport on a tree of the Walters variety, which is a white, seedy type. The Foster, discovered in 1906, is also a seedy type, and was the first pigmented grapefruit variety of record in Florida (Webber and Batchelor, 1943).

Hodgson (1955) said that the Thompson (Marsh Pink) originated as a limb sport on a Marsh tree in Florida, was discovered in 1913, and introduced in 1924. The pink color in the Thompson is limited to the pulp; that is, it shows no color in the rind as indicated by the absence of color in the membranes, mesocarp, and external fruit surface. The Thompson, in common with the Marsh, is nearly seedless and, according to Hodgson (1967), it was the first seedless pigmented variety to be discovered. In the early 1930's, the Thompson was widely planted in Texas but, as predicted by Webber (1943), this variety is likely to be superseded by the deeper pink and red varieties unless it still proves to be superior in quality.

The Ruby (Henninger's Ruby), of deep flesh color and with a red blush on the peel, likewise occurred as a limb sport on a Thompson tree in Texas, and was found in 1929. This fruit has characteristics similar to those of Thompson (Marsh Pink) in all respects, except for the deep-red color uniformly distributed throughout the membranes and pulp (juice vesicles) but not present in the juice. The fruit has colored albedo and a red blush on the exterior surface of the rind. The color of the flesh fades with fruit maturity. In Texas, Ruby is the most prominent pigmented variety, and has been extensively planted there.

The Webb (Webb's Redblush) is another mutant (limb sport) of the Thompson, discovered in 1931. As Webber (1943) noted, the Redblush is so similar to the Ruby that is is difficult to distinguish these varieties by physical appearance, because the Redblush also has a red blush on the outer rind, and deep-red flesh. The Thompson, Ruby, and Webb are nearly seedless, which is important with respect to their commercial possibilities. Of these 3 kinds, the Ruby has successfully superseded the Thompson, and has become the most prominent pigmented variety in commercial production.

The Burgundy (Burgundy Red) grapefruit is a late-maturing variety that was discovered in Florida about 1948. It is seedless, has red pulp, but fails to

develop external rind coloration under Florida growing conditions. The pulp of the Burgundy is more highly colored than that of the Redblush, and the color is retained over a longer maturation period. The Burgundy variety first occurred as an entire tree, which indicates that it originated as an undetected bud sport of the Thompson. The nucellar seedlings of the Burgundy do not produce the true color of the parent clone.

Varieties Grown in Various Areas. The following list gives the varieties and types of grapefruit grown in different parts of the world. The information concerning those grown in the United States was obtained from local publications; that from other areas, through personal correspondence.

Grown Commercially in the United States

Arizona .....Marsh Seedless; pigmented types: Redblush or Ruby Red.
California .....Marsh Seedless; pigmented types: Redblush or Ruby Red.
Florida.....Marsh Seedless, Duncan seedy; pink seedless: Redblush or Ruby Red.
Texas .....Marsh Seedless and Redblush.

Grown, Commercially or Not, in Other Areas

Algeria .....Marsh.
Argentina .....Duncan, 20 per cent; Marsh, 75 per cent; others, 5 per cent. (From Buenos Aires.)
Australia .....Marsh, Triumph. (From Brisbane.)
Brazil .....Marsh.
Br. Honduras ....Duncan, 10 per cent; Marsh, 90 per cent.
Cuba .....Duncan, Marsh.
Cyprus .....Marsh.
Israel .....Marsh.
Jamaica .....Duncan, Marsh, Silver Cluster.
Mexico .....Both white and pigmented varieties.
Mozambique ....Ellen, Marsh, Triumph.
Puerto Rico .....Duncan, Marsh.
Rhodesia (S.) ....Marsh, Triumph.
South Africa .....Marsh, Triumph (seedy type, decreasing in production). (From Pretoria.)
Spain .....Marsh Seedless.
Surinam .....Duncan (few), Marsh.
Trinidad-Tobago.Marsh.
Turkey .....Marsh (Marsh White), Thompson (Marsh Pink).
Uruguay .....Duncan, Marsh, some Foster and Thompson. Montevideo (Salto), mostly Duncan or Marsh.

In addition to the foregoing, Indonesia, the Philippines (Manila), French Cameroon, and French Equatorial Africa report that several varieties have been or are being tested, but that none has reached the commercial stage of development. Guadeloupe (F.W.I.), Chile (Santiago), and Ivory Coast (F.W. A.) report scattered plantings of several trees, but none on a commercial basis.

Returns from Five Varieties in Texas. Alderman (1951) made tests to determine the relative production and monetary value of 5 varieties of grapefruit growing in the Lower Rio Grande Valley of Texas. These tests were made to help the grower to decide which varieties to plant in place of those that were killed by the severe freezes of 1949 and 1951. The results are shown in table 1, which shows that Thompson (Marsh Pink) produced the highest average return per acre (0.405 hectare): 410 boxes and $623.20. Ruby brought $2.16 per box, which was the highest for all varieties. The percentage of total production of pink- and red-fleshed grapefruit to reach the market increased during the period of these tests (1940–50).

Production and Utilization of the World's Supply

THIS CHAPTER is principally concerned with the growth and development of grapefruit production in the various citrus-producing areas of the world. Many scientific and technological advances have occurred simultaneously with the enormous increase in world production. These advances have had their effect on the growing and utilization of grapefruit, and so have brought about many economic changes affecting both the grower and the consumer. Considerable attention is given here to these trends in the industry in the United States as well as in other producing areas, with due regard to exports. An attempt has been made to demonstrate the application of recent scientific developments to the utilization of the crop, developments that have resulted in a continued shift in the proportions used in fresh and processed forms. From the consumer's viewpoint, many forms of citrus fruit can be readily substituted for one another. Furthermore, among many fruits, such as oranges and grapefruit, there is a high degree of competition between fresh and processed forms of the same commodity.

The processing of citrus commodities is, to a varying degree, complicated and specialized; consequently, growers would find it difficult to process their own produce. During the past 40 years, the technological developments that have raised the level of productivity in food processing are those discoveries that have made it possible to transfer processing methods from batch to continuous operation. This continuity offers many obvious and marked advantages, such as reduction in cost by elimination of hand labor and less efficient machines. A product of higher quality accordingly results, by reducing the time during which it is exposed to contamination and the deteriorating effects of air and heat. An outstanding example of a new machine which made it possible to produce better and cheaper citrus products was the automatic juice extractor. The successful production in large volumes of canned single-strength and frozen concentrated citrus juices was made possible by the commercial application of automatic juice extractors. In the extraction of juice

TABLE 1

TEN-YEAR AVERAGES OF PRODUCTION RECORDS AND MONETARY RETURNS FOR 5 VARIETIES OF GRAPEFRUIT, 1940–50.*

Variety	Varietal ratio (per cent)	Average yearly juice production (1.6-bushel boxes)†	Average yearly fresh production (1.6-bushel boxes)	Average yearly total production (1.6-bushel boxes)	Average return per box (dollars)‡	Average production per acre (boxes)	Average return per acre (dollars)
Marsh White	71.6 fresh 84.4 juice	6,091,823	7,281,863	13,373,686	1.08	366	395.28
Marsh Pink	16.4 fresh 7.4 juice	534,117	1,667,913	2,202,030	1.52	410	623.20
Foster Pink	6.9 fresh 3.4 juice	245,405	701,744	947,149	1.62	337	545.94
Ruby Red	4.9 fresh 2.2 juice	158,792	498,340	657,132	2.16	135	291.60
Duncan	0.2 fresh 2.6 juice	187,663	20,340	208,003	0.78	310	241.80
Totals	100.0 fresh 100.0 juice	7,217,800	10,170,200	17,388,000			

*Adapted from Alderman (1951).
†One bushel equals about 35.24 liters.
‡Weighted averages.

from oranges and grapefruit, the transfer of energy from manpower to machine power has resulted not only in enormous savings in labor cost, but in corresponding increases in the amount and quality of juice produced.

The rapid advances made in the pasteurization of liquid foods were important factors in the successful production of citrus juices with respect to quality in terms of stability and flavor. Citrus juices were formerly pasteurized by the batch method in kettles, or by heating after canning at relatively high temperatures for 5 to 30 minutes. Such sterilization procedures altered flavor and quality. With the development of flash pasteurization, the juices were rapidly heated in tubular or plate heaters to a high temperature below the boiling point of water for a few seconds, and then filled hot into final containers which were afterward rapidly cooled. An improvement on the flash-pasteurization method was commonly known as the "high-short" sterilization procedure; actually, the high-temperature short-time method (HTST), which consisted of rapidly heating the juices to temperatures above 212° F. (100° C.) for a few seconds, followed by rapid cooling and filling into sterile containers under aseptic conditions. This procedure indicates that the higher the temperature, the shorter the time required for sterilization.

These are only a few of the technological advances that have contributed to the successful processing of citrus fruits into high-quality juice products. Such outstanding technical developments in juice processing have been briefly noted for the purpose of showing ways to increase the efficiency of processing by constantly improving the automatic instrumentation and technological developments so essential to secure high-quality citrus products.

World Production. During the 6-year period beginning in 1963–64, the average annual world production of grapefruit was 60.4 million boxes, and in 1964 and 1965, it was 53.2 and 60.6 million boxes, respectively—a considerable increase over the 1963–64 average (table 2). The estimated world total for 1968–69 was 72.2 million boxes, a 66 per cent increase over the crop season 1963–64, but also significantly above the 1967–68 average value. The United States production for 1968–69 was approximately 55.9 million boxes, 21.5 million boxes more than in 1963–64. This accounted for 77.4 per cent of the total world production.

Israel was second to the United States in the production of grapefruit for the crop season 1968–69 (table 2). Production has been on the increase for several years, and the upward trend will, in all probability, continue on through the 1970's. The 1965–66 production figures were nearly a million boxes in excess of those for 1964–65.

The table also shows that other countries in the Northern Hemisphere, such as Jamaica, Trinidad-Tobago, Cyprus, and Morocco, have been experiencing upward trends in grapefruit production, and record new highs are predicted for these producing areas.

TABLE 2

PRODUCTION OF FRESH GRAPEFRUIT IN SPECIFIED COUNTRIES OR AREAS IN THE CROP SEASONS 1963–64 THROUGH 1968–69.*

Countries or areas	Crop seasons (production in million boxes)†					
	1963–64	1964–65	1965–66	1966–67	1967–68	1968–69
North America						
British Honduras	0.3	0.2	0.2	0.2	0.2	0.3
Jamaica	.7	.7	.7	.5	.7	.6
Mexico	.2	.2	.3	.3	.3	.3
Trinidad–Tobago	.6	1.0	.8	.7	.5	.5
United States	34.4	41.7	47.4	57.4	44.5	55.9
Totals	36.2	43.8	49.4	59.1	46.2	57.6
Mediterranean area						
Algeria	0.1	0.1	0.1	0.1	0.1	0.1
Cyprus	.4	.6	.7	.9	1.0	1.0
Israel	3.6	4.4	5.1	6.1	7.3	7.3
Morocco	.3	.3	.4	.5	.4	.4
Spain	.1	.1	.2	.2	.2	.2
Totals	4.5	5.5	6.5	7.8	9.0	9.0
Northern Hemisphere Totals	40.7	49.3	55.9	66.9	55.2	66.6
South America						
Argentina	1.4	1.7	2.0	2.3	2.3	2.3
Surinam	.2	.1	.1	.2	.2	.2
Totals	1.6	1.8	2.1	2.5	2.5	2.5
Other Southern Hemisphere						
Australia	0.2	0.2	0.3	0.2	0.3	0.2
New Zealand	.1	.1	.1	.1	.1	.1
South Africa	1.5	1.8	2.2	2.2	2.4	2.8
Totals	1.8	2.1	2.6	2.5	2.8	3.1
Southern Hemisphere Totals	3.4	3.9	4.7	5.0	5.3	5.6
Totals, specified countries	44.1	53.2	60.6	71.9	60.5	72.2

*From USDA (1965, 1969*a*). The data have been prepared or estimated on the bases of official statistics of foreign governments, other foreign source materials, reports of United States agricultural attachés and Foreign Service officers, results of office research, and related information.

†Harvests in the Southern Hemisphere begin in the spring of the year shown, and those in the Northern Hemisphere begin in the fall. All production figures have been converted to equivalents of millions of boxes of 80 pounds (about 36 kilograms) each.

South Africa and Argentina are the largest producers of grapefruit in the Southern Hemisphere. In fact, these countries produced 5.1 million boxes in the crop season 1968–69 out of a total of about 5.6 million, which was equivalent to 91.1 per cent of the total production for the hemisphere. It should be stated that the figures quoted for Argentina include unharvested production and fruit not of export variety or grade.

The Northern Hemisphere accounted for 92.2 per cent of the world's total production—66.6 million boxes out of a total of 72.2 million.

The variations in production from year to year in the respective countries are shown in the annual yields for the 6 crop seasons, 1963–64 through 1968–69. Some of this variation was caused by weather conditions, such as frost during the blooming period, rainfall, and partial loss of the preharvest crop by freezes. The steady trend in increased production in other cases was largely because of young trees coming into bearing, as well as some increase in new plantings. This factor has been especially important in North America and the Mediterranean area. In fact, continued increases in the production of grapefruit are expected as a result of new plantings which will come into bearing in the United States, the Mediterranean area, South America, and South Africa.

Trends in Production and Consumption in the United States

THE COMMERCIAL BEARING acreage is shown in table 3. Florida, Texas, California, and Arizona are the 4 grapefruit-producing areas in the United States. The total bearing acreage and total production of grapefruit in this country have shown considerable fluctuation since the 1935–36 crop season (not reported in table 3). The most revealing fact about the table is that Florida has the largest bearing acreage, and consequently the highest production of grapefruit in the United States. The state reached its peak in bearing acreage in the crop season 1956–57, with a declining trend to 83,000 acres for 1963–64, increasing slightly thereafter. Texas plantings reached a maximum of 82,000 bearing acres in 1948–49 (not reported in table 3), and this diminished to 38,100 acres for 1959–60, which is the last entry recorded for this area. California has never had over 16,500 acres (1937–38), and this declined to 7,200 for the crop season 1956–57. In 1964–65, California had 11,900 bearing acres, while Arizona was reported to have 6,100 bearing acres—slightly lower than the 7,000 figure quoted for 1935–36. In the crop season 1935–36, Florida had 44.9 per cent of the total bearing acreage in the United States and, in 1968–69, this amount had increased to about 66 per cent.

Nonbearing Acres. A knowledge of the relationship of bearing acreage to nonbearing enables one to forecast within some limitations the probable

TABLE 3

UNITED STATES GRAPEFRUIT-BEARING ACREAGE AND PRODUCTION FIGURES, REPORTED BY STATES FOR THE CROP SEASONS 1951-52 THROUGH 1968–69.*

Crop seasons	Florida		Texas		California		Arizona		United States	
	Bearing acreage†	Production of value†	Bearing acreage	Production of value	Bearing acreage	Production of value	Bearing acreage	Production of value	Bearing acreage	Production of value
1951–52	98.6	33,000	17.9	200	9.7	2.160	8.9	2,140	135.1	37,500
1952–53	102.2	32,500	20.9	400	9.4	2,458	8.5	3,000	141.0	38,358
1953–54	105.5	40,700	22.0	1,200	9.1	2,500	7.0	2,670	143.6	47,070
1954–55	111.2	34,800	24.1	2,500	8.2	2,414	6.0	2,470	149.5	42,184
1955–56	111.8	38,300	26.0	2.200	8.3	2,507	6.0	2,370	152.1	45,377
1956–57	112.4	37,400	29.7	2,800	7.2	2,407	5.5	2,180	154.8	44,787
1957–58	95.0	31,100	30.1	3,500	7.5	2,397	5.4	2,780	138.0	39,777
1958–59	94.0	35,200	34.1	4,200	7.4	2,527	5.6	1,870	141.1	43,797
1959–60	92.3	30,500	38.1	5,200	7.6	2,671	5.6	3,220	143.6	41.591
1960–61	92.5	31,600	....‡	6,800	8.1	2,630	5.7	2,340	.....	43,370
1961–62	94.0	34,800	....	2,700	9.5	2,820	5.8	2,170	.....	42,490
1962–63	88.0	30,000	....	70	10.9	2,498	6.0	2,170	.....	34,738
1963–64	83.0	26,300	....	500	11.9	4,197	6.0	3,210	.....	34,207
1964–65	84.0	31,900	....	2,000	11.9	4,227	6.1	2,900	.....	41,027
1965–66	86.0	34,900	....	3,800	12.0	4,945	6.2	3,050	.....	46,695
1966–67	87.0	43,600	....	5,400	12.8	4,996	6.6	1,680	.....	55,676
1967–68	87.5	32,900	....	2,800	12.8	4,618	6.7	3,740	.....	44,058
1968–69	91.2	39,900	27.5	6,700	12.8	4,960	5.1	2,510	136.6	54,070

*From Florida Department of Agriculture (1969).
†Acreage and value figures are stated in thousands (of acres or boxes). One acre equals about 0.405 hectare.
‡Blanks mean "data not available."

production trends of the future (table 4). In 1964, Florida had 5.6 million bearing trees and 660,000 nonbearing trees, which was equivalent to about 10,200 acres of nonbearing grapefruit. The number of nonbearing trees amounted to 11.8 per cent of the total number of bearing ones (5.6 million). In terms of acreage, the number of nonbearing acres also amounted to 11.8 per cent of the total bearing acres (86,200 in 1964). If the total number of bearing trees (5.6 million) is divided by the total bearing acres (86,200), the quotient is the total number of trees per acre (65 trees). Even though grapefruit trees are relatively long-lived, a certain percentage of nonbearing acreage continuously passing into the bearing class each year is necessary to keep up production in any given district. As of December, 1967, there existed in Florida 32,366 acres of nonbearing grapefruit trees less than 4 years old; and, in 1969, grapefruit constituted the next largest segment, with 91,178 acres, which amounted to 11.9 per cent of the bearing commercial citrus acreage.

Grapefruit Production by States. Commercial production by states, 1951–52 through 1968–69, is shown in table 3. On the basis of the data presented, Florida produced 78.1 per cent of the total United States crop in the crop season 1964–65. Through the years, Florida has been the leading grapefruit-producing state, followed by Texas, with California and Arizona being about equal in production. For example, between 1935 and 1945 the production of grapefruit in the United States increased from 18 million to 63 million boxes (figure 1). Improvements in transportation, storage, and processing have been important factors in causing this large increase (Harding, 1947*a, b*). Reitz (1946) stated that during the 14 years preceding 1946, the production in Florida increased 199 per cent. Between 1935–36 and 1964–65, production in that state increased from 11.5 million boxes to 31.9 million boxes.

The data in figure 1 show graphically the relative changes in grapefruit production in the United States with reference to the amounts produced in Florida, Texas, California, and Arizona for a 30-year period. The total nationwide production reached its peak in the 1945–46 crop season. Production declined rapidly to 1949–50, rose significantly in 1950–51, and has subsequently remained at an average level with sizable annual fluctuations. It should be pointed out that Texas also reached its peak in 1945–46, followed by a downward trend through 1962–63. This enormous reduction in grapefruit productivity in Texas was caused chiefly by a series of severe freezes that occurred in 1949–50 and 1950–51, resulting in a low of 200,000 boxes for the 1951–52 crop season; by 1959–60, the output had increased to 5.2 million boxes. However, this recovery was interrupted by another severe freeze in January, 1962, which reduced the figures for 1962–63 to 70,000 boxes. This downward trend for Texas since 1945–46 is reflected in the curve for total nationwide production. California and Arizona show an increase after 1960. After 1936, Florida had an upward trend.

TABLE 4

THE NUMBER OF BEARING AND NONBEARING GRAPEFRUIT TREES IN FLORIDA DURING 3 REPRESENTATIVE YEARS.*

Type of fruit	Nonbearing trees			Bearing trees			Total trees		
	1957	1963	1964	1957	1963	1964	1957	1963	1964
Seedy	108†	70	60	2,467	1,640	1,700	2,575	1,710	1,760
Seedless	599	420	600	4,228	3,670	3,900	4,827	4,090	4,500
Totals, both types	707	490	660	6,695	5,310	5,600	7,402	5,800	6,260

*Adapted from Florida Department of Agriculture (1965).
†All figures are stated in thousands of trees.

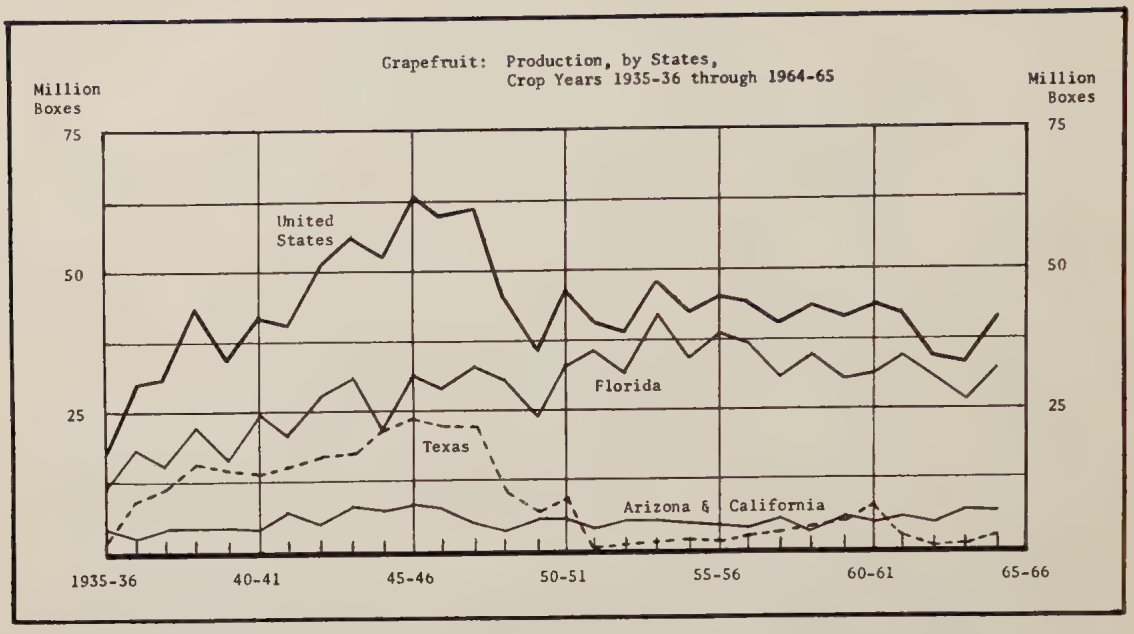


Fig. 1. Grapefruit production by states in the crop seasons 1935–36 through 1964–65. (From Florida Department of Agriculture, 1965.)

Utilization of Grapefruit. An estimate of the utilization of the Florida crop from 1935–36 through 1968–69 is given in table 5. This shows the great changes in utilization that have taken place concurrently with the large increase in the total crop.

The continuing tendency to make use of increasingly more of the crop for processing into canned and frozen products was born of the necessity to market increasingly larger annual crops.

The data in table 5 that show the relationship of production to utilization of the crop from 1935–36 through 1964–65 are revealing, in that during that period the volume of fresh fruit shipped by rail decreased, with a concomitant increase in the amount shipped by truck. Truck shipments, for the first time, exceeded those by rail in 1954–55, and this trend is still on the increase. With expanded facilities in the field of truck transportation, such as better highways and more equipment, and with the increased use of more efficient refrigeration, this method of transporting perishable fruits will continue to be highly favored by shippers. Boat shipments of fresh grapefruit have been on a decided decrease for many years.

Speed of handling and transportation is an important factor in determining the type of carrier used for transporting perishable products. The rate of speed with which fresh fruits and vegetables reach the market is highly correlated with the rate of spoilage and the fluctuating prices that are constant elements of risk in the marketing of these short-lived commodities.

It should be noted that large quantities of commercial perishable fruits, such as citrus, are produced in areas far from the large eastern and midwestern

TABLE 5

FLORIDA GRAPEFRUIT: BEARING ACREAGE, PRODUCTION, AND UTILIZATION IN THE CROP SEASONS 1935–36 THROUGH 1968–69.*

Crop seasons	Bearing acreage†	Total production‡	For farm home use	Sold for fresh use out of state				Sold for fresh use in Florida	Totals sold for fresh use	Sold for processing
				Rail	Boat	Truck	Total			
1935–36	77.0	11,500	92	3,419	2,542	1,031	6,992	770	7,762	3,646
1936–37	78.7	18,100	108	6,416	3,056	936	10,408	825	11,233	6,759
1937–38	80.4	14,600	94	4,433	2,196	850	7,479	870	8,349	6,157
1938–39	81.5	23,300	143	6,653	3,204	1,398	11,255	971	12,226	9,212
1939–40	82.5	15,900	90	3,550	1,518	980	6,048	950	6,998	8,812
1940–41	82.5	24,600	100	5,743	2,208	1,631	9,582	1,042	10,624	13,876
1941–42	82.8	19,200	101	6,646	206	984	7,836	1,120	8,956	10,143
1942–43	83.6	27,300	113	7,999	.....	521	8,520	1,083	9,603	17,584
1943–44	84.1	31,000	118	8,953	.....	356	9,309	1,127	10,436	20,446
1944–45	85.5	22,300	105	5,860	.....	222	6,082	977	7,059	15,136
1945–46	85.2	32,000	140	8,297	10	340	8,647	1,077	9,724	22,136
1946–47	85.7	29,000	120	8,248	631	558	9,437	977	10,414	15,866
1947–48	87.6	33,000	140	7,486	9	1,143	8,638	1,071	9,709	19,451
1948–49	89.5	30,200	140	8,827	.....	3,624	12,451	1,303	13,754	16,306
1949–50	93.0	24,200	140	4,965	827	3,565	9,357	1,214	10,571	13,489
1950–51	94.4	33,200	150	9,071	12	4,798	13,881	1,316	15,197	17,853
1951–52	98.6	36,000	150	10,686	84	6,830	17,600	1,572	19,172	13,678
1952–53	102.2	32,500	160	8,572	31	7,189	15,792	1,513	17,305	15,035
1953–54	105.5	42,000	160	9,601	100	9,254	18,955	1,496	20,451	20,089
1954–55	111.2	34,800	160	8,154	42	9,040	17,236	1,760	18,996	15,644
1955–56	111.8	38,300	160	7,615	132	9,990	17,737	1,745	19,482	18,658
1956–57	112.4	37,400	160	6,640	124	9,645	16,409	1,778	18,187	19,053
1957–58	95.0	31,100	160	5,085	98	7,727	12,910	1,634	14,544	16,396
1958–59	94.0	35,200	160	5,527	119	9,067	14,713	1,766	16,479	18,561
1959–60	92.3	30,500	160	4,808	38	9,209	14,055	1,977	16,032	14,308
1960–61	92.5	31,600	160	4,808	113	8,827	13,748	1,978	15,726	15,714
1961–62	94.0	35,000	180	4,989	274	10,569	15,832	1,979	17,811	16,809
1962–63	87.7	30,000	125	2,707	115	9,244	12,066	1,847	13,913	15,962
1963–64	81.6	26,300	180	2,848	65	9,634	12,547	1,992	14,539	11,581
1964–65	86.2	31,900	180	3,940	25	9,596	13,561	2,105	15,666	16,054
1965–66	86.0	34,900	180	.....§	.....	.....	12,812	2,085	14,897	19,823
1966–67	87.0	43,600	180	.....	.....	.....	14,956	2,145	17,101	26,319
1967–68	87.5	32,900	180	.....	.....	.....	12,553	1,969	14,522	18,198
1968–69	91.2	39,900	175	.....	.....	.....	12,367	1,525	13,892	25,833

*From Florida Department of Agriculture (1965, 1969).
†Stated as thousands of acres. (One acre equals about 0.405 hectare.)
‡Includes economic abandonment. (All production figures are in thousands of boxes of 80 pounds, or about 36 kilograms, each.)
§Blanks mean "data not available."

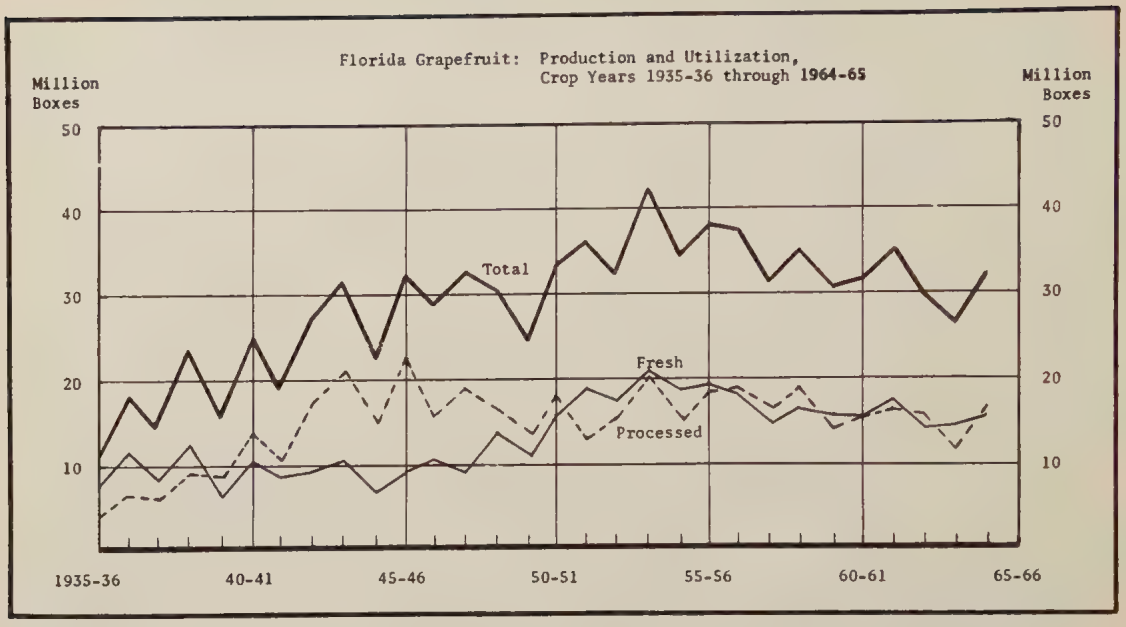


Fig. 2. Florida grapefruit: production and utilization in the crop seasons 1935–36 through 1964–65. (From Florida Department of Agriculture, 1965.)

terminal markets. In order that a salable, high-quality product may reach the consuming public, the application of considerable technological skill to transportation problems is definitely required.

Another relationship that is clearly indicated in table 5 shows the fractions of the total crop that are sold either for fresh use or for processing purposes. The course of these changes for 30 and 25 years, respectively, is delineated in figures 2 and 3. Between the crop seasons 1935–36 and 1939–40, a higher proportion of the total crop was sold for fresh use; from 1939–40 through 1950–51, the trend was reversed, and a higher proportion of the total was sold for processing purposes. From 1950–51 through 1964–65, the total crop was about equally distributed (50 per cent) between these 2 marketing channels. The data in figure 3 show distinctly that from the crop season 1964–65, the trend in the volume of fruit processed was on the increase. With continued expansion in world production of grapefruit, additional uses other than fresh sales will undoubtedly be found through research. Citrus growers recognize the difficulties involved in marketing huge stocks of grapefruit, both domestically and internationally, through fresh-market channels, and consequently are convinced that processing is their only alternative outlet. It appears that more and more fruit will be diverted to that medium.

The total production of Florida grapefruit is composed of both the seedy and seedless types. The utilization of the crop with respect to the volume of fruit diverted to either the fresh-fruit consumers or to processed products is compared by types for the crop seasons 1945–46 through 1968–69 in figures 4, 5, and 6. During this period, the seedy type of grapefruit showed a down-

ward trend in production from about 18 to 12.2 million boxes, while the pink seedless type showed an erratic increase from 4.7 to 10.7 million boxes from 1954–55 through 1968–69 (figure 5). In the crop season 1954–55, the production of white seedless grapefruit amounted to 15.8 million boxes, followed by a downward trend to a minimum of 11.9 million boxes for the crop season 1960–61, after which an upward trend to 17 million boxes for 1968–69 appeared (figure 6). During 1968–69, the production of all 3 types was 39.9 million boxes, which represented the total output of Florida grapefruit.

Between 1945–46 and 1953–54, the production of seedy grapefruit was marked only by seasonal fluctuations (figure 4). During the 15-year period, 1953–54 through 1968–69, the total production of seedy grapefruit declined from 19.1 to 12.2 million boxes, a decrease of 6.9 million boxes. In the seedy type, the quantity sold for processing amounted to about 11.4 million boxes for 1968–69, and the volume for fresh use was 767,000 boxes, excluding 45,000 boxes disposed of for farm home use. From these data, it can be computed that 93.4 per cent of the total production of the seedy type (crop season 1968–69) was utilized in processed forms. Since 1951–52, the volume of seedy grapefruit marketed through fresh-fruit channels has been on a slow but steady decline (see the curve marked "fresh" in figure 4). It is worthy of note that, in addition to the gradual decline in total production of seedy grape-

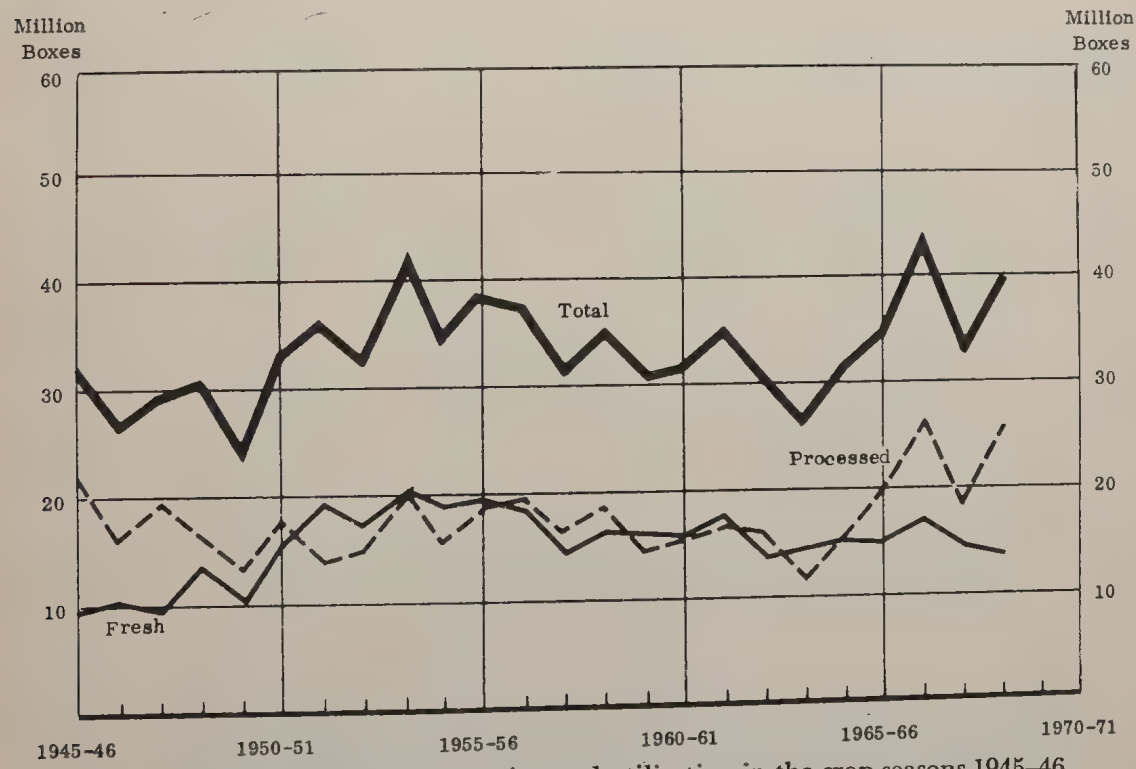


Fig. 3. Florida grapefruit: production and utilization in the crop seasons 1945–46 through 1968–69. (Florida Department of Agriculture, 1969.)

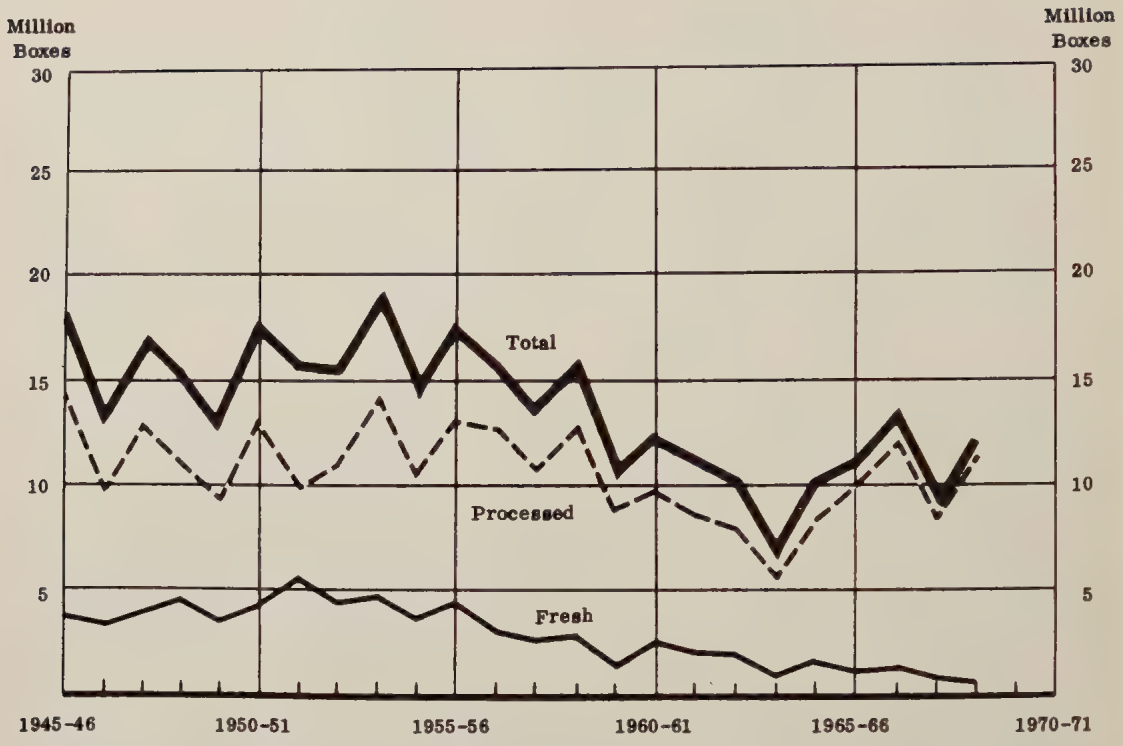


Fig. 4. Florida seedy grapefruit: production and utilization in the crop seasons 1945–46 through 1968–69. (From Florida Department of Agriculture, 1969.)

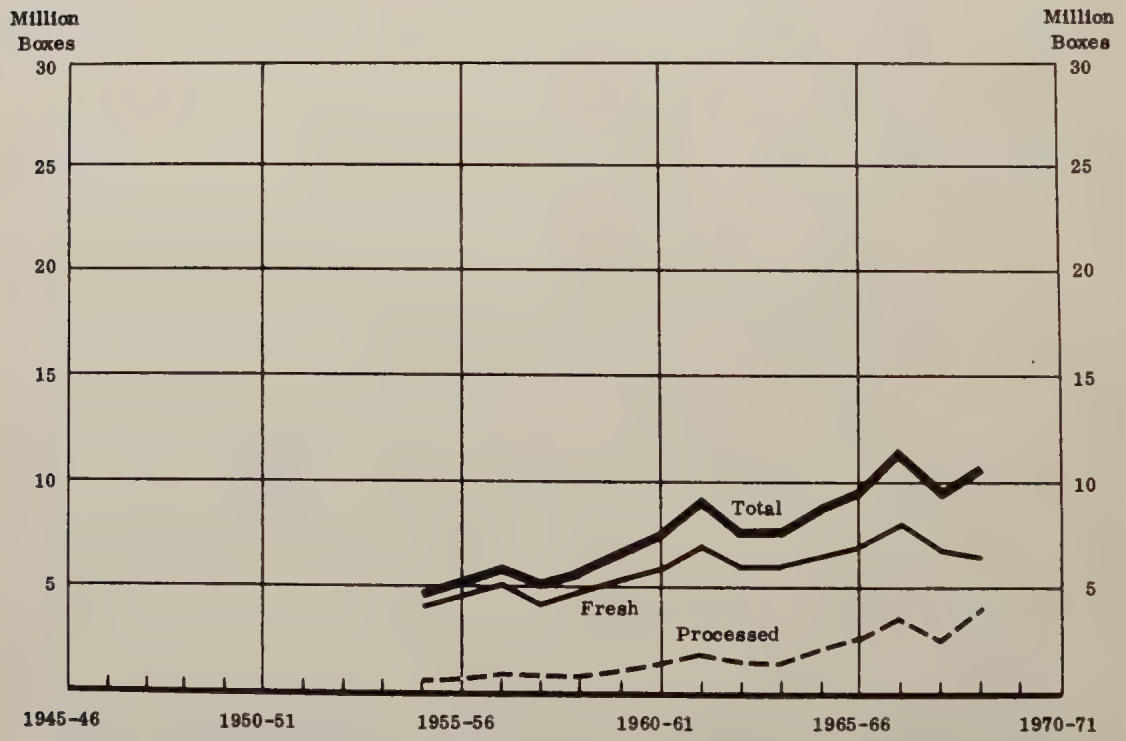


Fig. 5. Florida pink seedless grapefruit: production and utilization in the crop seasons 1954–55 through 1968–69. (From Florida Department of Agriculture, 1969.)

fruit, the bearing acreage also steadily declined during the same period up to the 1960–61 crop season, after which it became stabilized at approximately 25,000 acres.

During 1968–69, the total production of Florida pink seedless grapefruit (10.7 million boxes) was utilized as follows: 6.6 million boxes were sold for fresh use, and 4.1 million boxes were sold for processing purposes. The quantity of fruit used for processing amounted to 38.3 per cent of the total production (figure. 5).

In the utilization of white seedless grapefruit, the proportion of the crop sold for fresh-fruit use and for processing has varied considerably since 1954–55 (figure 6). The trend in the amounts of this type used in fresh form was on a continuous decline from 1954–55 to 1968–69, and during this 14-year period the amounts of fruit used for processed products increased from 4.4 to 10.4 million boxes.

It can be observed that the commercial production of Florida grapefruit has been mainly distributed among 3 types: white seedless, pink seedless, and seedy (table 6). The highest quality of grapefruit is grown in the Indian River section. In the crop season 1968–69, the production of white seedless amounted to 17 million boxes, of which 5.3 million (31.2 per cent) came from the Indian River area. The state production of pink seedless was 10.7 million boxes, of which 4.5 million (42 per cent) were produced in the Indian

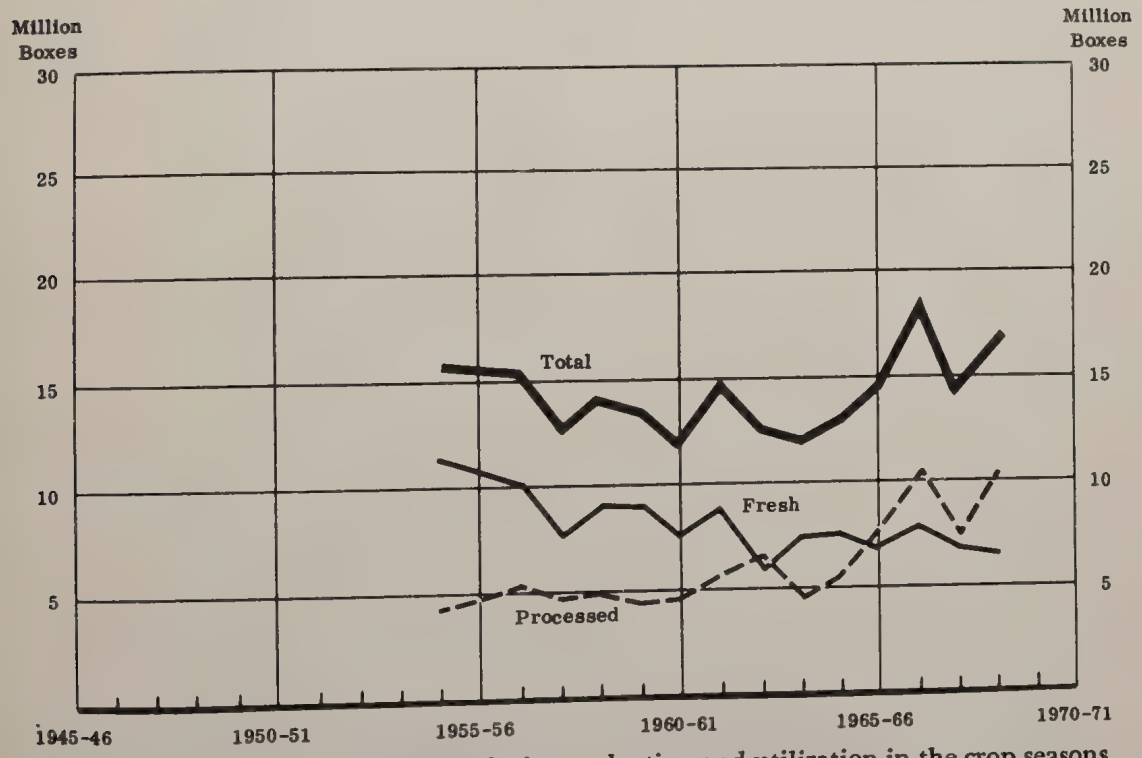


Fig. 6. Florida white seedless grapefruit: production and utilization in the crop seasons 1954–55 through 1968–69. (From Florida Department of Agriculture, 1969.)

TABLE 6

FLORIDA GRAPEFRUIT: PRODUCTION BY TYPES AND BY AREAS OF PRODUCTION IN THE CROP SEASONS 1960–61 THROUGH 1968–69.*

Crop seasons	White seedless			Pink seedless			Seedy		
	State	Interior	Indian River	State	Interior	Indian River	State	Interior	Indian River
1960–61	11,900†	7,800	4,100	7,300	3,500	3,800	12,400	11,800	600
1961–62	14,700	10,900	4,800	9,000	4,700	4,300	11,100	10,500	600
1962–63	12,500	8,000	4,500	7,500	3,100	4,400	10,000	9,400	600
1963–64	12,100	6,800	5,300	7,600	2,400	5,200	6,600	5,900	700
1964–65	13,000	9,100	3,900	8,700	4,500	4,200	10,200	9,800	400
1965–66	14,400	9,600	4,800	9,300	4,700	4,600	11,200	10,600	600
1966–67	18,600	13,700	4,900	11,500	6,400	5,100	13,500	13,100	400
1967–68	14,300	9,200	5,100	9,400	4,400	5,000	9,200	8,950	250
1968–69	17,000	11,700	5,300	10,700	6,200	4,500	12,200	11,900	300

*From Florida Department of Agriculture (1969).
†Production figures are stated as thousands of boxes.

River area. The seedy type was mainly grown in the interior part of the state (12.2 million boxes), only 300,000 boxes being produced in the Indian River section. The total output of these 3 types was 39.9 million boxes, which was equivalent to the amount of Florida grapefruit marketed in both fresh and processed forms.

Canned Juices and Segments. Research and development in citrus-fruit processing originally began in the United States for the purpose of diverting excess fruit from fresh-fruit marketing channels in order to stabilize prices. Processing methods and proper storage facilities have permitted expanded use of production possibilities in areas having seasonal disadvantages, thus playing an important role in increasing the world's food supply. With the growth of the food-processing industries, spectacular results have been achieved in producing the readily available canned grapefruit juices, frozen concentrates, and grapefruit segments.

The development and commercial production of frozen citrus concentrates (especially orange) form a notable example of the development of a product in a new form which can satisfactorily compete with fresh fruit and with other ways of processing citrus juice. As noted in connection with figures 2 and 3, approximately 50 per cent of the Florida grapefruit crop is processed. With the continued diversion of such a large proportion of the crop to processed products, it is also imperative that continued improvement be maintained in their quality. From the consumer's viewpoint, many fruits can be readily substituted for one another. In fact, with citrus fruits such as grapefruit, a high degree of competition exists between the fresh and processed forms of the same commodity. This competitive situation may cause further changes in the relative amounts of grapefruit used in the fresh, canned, and frozen concentrate markets.

Table 7 shows an estimate of the annual output of canned grapefruit products by states in equivalent cases of 24 No. 2 cans for the crop years 1941–42 through 1964–65. Florida is by far the largest processor of grapefruit in the United States, and in 1964–65 the state appears to have been the only producer of canned grapefruit segments in the country. For the crop year 1963–64, Florida produced 81.6 per cent of the total single-strength grapefruit juice packed in the United States. According to the table, California and Arizona accounted for the remaining 18.4 per cent, which amounted to approximately 1.16 million cases. No figures were recorded for Texas. Florida was also the chief producer of single-strength blended juice in the country. The United States estimated pack of single-strength grapefruit juice and segments amounted to 18.6 million cases in 1956–57 and to 9.4 million cases in 1963–64.

Production in Texas was reduced during this period by freeze damage to both trees and fruit.

TABLE 7

ANNUAL PRODUCTION OF CANNED GRAPEFRUIT SEGMENTS AND JUICE, IN EQUIVALENT CASES OF 24 NUMBER 2 CANS, REPORTED BY STATES FOR THE CROP SEASONS 1941–42 THROUGH 1964–65.*

Crop seasons	Grapefruit segments				Single-strength grapefruit juice				Single-strength blended juice				Citrus salad‡
	Florida	Texas	California and Arizona	United States	Florida	Texas	California and Arizona	United States	Florida	Texas	California and Arizona	United States	
1941–42	4,611†	90	...	4,701	6,180	5,508	1,095	12,783	2,305	38	0	2,343	2
1942–43	888	87	...	975	15,193	7,360	1,049	23,602	3,676	25	1	3,702	0
1943–44	943	41	...	984	16,778	7,864	1,450	26,092	6,176	69	38	6,283	0
1944–45	411	54	...	465	12,025	9,144	1,266	22,435	7,745	254	76	8,075	0
1945–46	2,407	289	...	2,696	15,089	9,601	1,372	26,062	12,267	626	568	13,461	0
1946–47	5,098	490	...	5,588	8,583	8,240	791	17,614	10,034	340	85	10,459	310
1947–48	3,158	242	...	3,400	7,987	9,981	482	18,450	11,894	299	176	12,369	1,274
1948–49	4,238	189	...	4,427	8,843	4,944	517	14,304	10,252	229	348	10,829	1,098
1949–50	3,379	...§	...	3,379	7,894	2,680	1,633	12,207	6,768	45	582	7,395	433
1950–51	4,628	...	...	4,628	12,742	4,551	933	18,286	8,711	369	355	9,435	954
1951–52	3,396	...	...	3,396	8,735	....	595	9,330	6,402	...	302	6,704	611
1952–53	3,811	...	...	3,811	10,854	...	790	11,644	5,707	...	410	6,117	689
1953–54	4,332	...	...	4,332	14,882	...	727	15,609	6,402	...	123	6,525	875
1954–55	5,244	...	...	5,244	10,784	445	593	11,822	4,994	12	98	5,104	810
1955–56	4,759	...	...	4,759	12,805	337	510	13,652	5,265	9	114	5,388	719
1956–57	4,518	...	...	4,518	12,464	1,173	456	14,093	5,188	24	90	5,302	591
1957–58	4,179	2	...	4,181	9,484	818	334	10,636	4,885	16	43	4,944	488
1958–59	4,572	...	...	4,572	10,093	1,064	227	11,384	4,212	32	109	4,353	591
1959–60	4,004	25	29	4,058	9,323	1,408	546	11,277	4,382	50	92	4,524	514
1960–61	4,326	25	...	4,351	9,131	1,509	522	11,162	3,101	93	...	3,194	356
1961–62	4,209	11	...	4,220	10,190	447	549	11,233	3,863	47	...	3,910	419
1962–63	2,613	0	...	2,613	8,864	0	551	9,415	3,117	0	13	3,130	88
1963–64	3,063	...	...	3,063	5,143	....	1,162	6,305	2,416	...	157	2,573	455
1964–65	3,606	...	...	...	9,770	...	...	...	2,435	...	...	...	301

*From Florida Department of Agriculture (1965).
†Production figures are stated as thousands of cases.
‡Includes orange segments.
§Blanks mean "data not available"; zeros mean "none produced."

Exports and Imports; Production Data

THE CITRUS INDUSTRY of the United States exported citrus fruits and products to Canada in substantial amounts before World War II, and this sizable volume has continued to increase in subsequent years. During this same period, the United States exported a considerable volume of fresh citrus fruit and products to the United Kingdom, but after the war this market almost disappeared, which later resulted in some increase of exports to Western European countries. The increased trend in United States grapefruit production that is predicted for the next few years should bring forth an increased stimulus for further expansion in the foreign market. The most important export outlet for fresh grapefruit and its products is the Western European market.

The economic prospects for increased foreign exports of citrus fruits and juice products can be realistically evaluated from the data in figure 7, which show the relationship between export and import values of United States trade in fruits, vegetables, and tree nuts for the 13-year period from 1956 through 1968. Although fresh citrus fruits and juice products are included in the calculations of these data, fruits other than citrus, such as imports of pineapple juice into the United States, may have a greater influence on the export:import ratios. Shipments of foreign fruits and vegetables have continued to expand during the last few years as a result of the relatively high

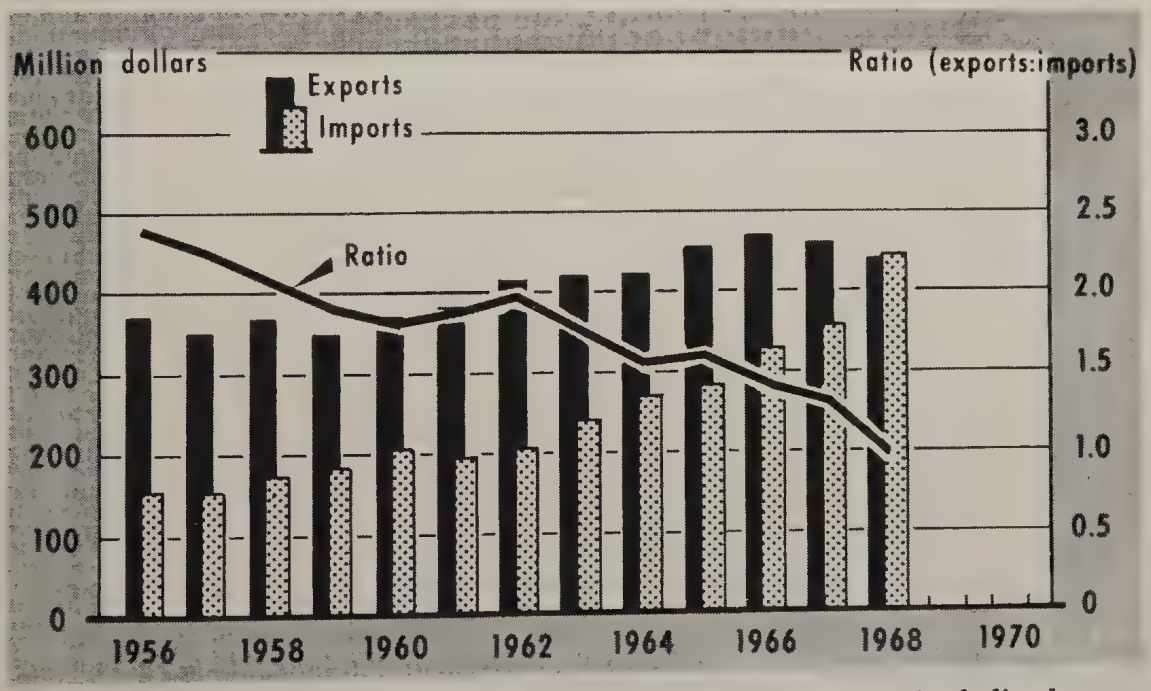


Fig. 7. Value of United States trade in fruits, vegetables, and tree nuts (excluding bananas and banana products), 1956 through 1968. Four prominent commodities: canned pineapple, peaches, and asparagus, together with fresh and processed citrus, accounted for 30 per cent of the total value of exports. (From Schroeter, 1969.)

monetary income per acre obtained from these commodities, increased demand in world markets owing to rising incomes, and the abundant supply of low-priced labor. Furthermore, foreign agricultural industries have developed and applied modern production and processing methods and marketing systems which have placed them in a strategic position to exploit successfully the inflationary tendencies in the United States and to erode gradually the competitive position of some important American export commodities (Schroeter, 1969). In the production and exporting of canned pineapple, Taiwan has contributed significantly to the increase of United States imports and to the decrease of United States export markets. This view is supported further by the fact that in 1956 the value of American exports of fruits, vegetables, and tree-nut crops was 2.5 times the value of imports. This ratio decreased in subsequent years to the position where, in 1968, our imports exceeded exports by more than 3 million dollars. Since 1962, the imports have increased gradually to a total of 442 million dollars in 1968, while exports declined 7 per cent to 439 million dollars.

This economic situation is partially applicable to United States exports of fresh citrus and citrus products to foreign markets. In terms of both volume and monetary value, oranges exceeded all other fresh fruits in these exports, which amounted to 334,000 short tons[1] in 1967. With reference to grapefruit, Israel is the chief competitor of American citrus exports of fresh grapefruit and citrus products to Western Europe, the second largest market for the United States. Israel, Italy, and Morocco are concentrating their efforts on increasing the export sales of citrus juices, thus creating greater competition against United States products in Western European markets. The development of citrus processing in the Mediterranean area, South Africa, and South America, for whatever reason, has forced these suppliers to find additional export markets, and the most receptive areas for their products have proved to be Western Europe and Canada, the leading outlets for our own exports.

United States Exports. The exports of fresh grapefruit from the United States to various other countries are shown in table 8. Annual shipments are reported for the 6-year period, 1962–63 through 1967–68. There has been a substantial increase in total exports of fresh grapefruit to Western Europe, extending from 1962–63 through 1966–67, with a subsequent decline in 1967–68. Total exports of the United States decreased from about 3.3 million boxes in 1966–67 to 2.3 million boxes in 1967–68, a reduction of approximately a million boxes. Most of the markets received less United States grapefruit in 1967–68, with Canada, by far the most important market, showing the largest decline in imports (over 660,000 boxes). The amount of fresh fruit exported to Europe in the crop season 1966–67 was 813,000 boxes, which was

[1] A "short" ton (2,000 pounds) equals about 908 kilograms. One pound equals 0.454 kilogram.

TABLE 8

EXPORTS OF FRESH GRAPEFRUIT FROM THE UNITED STATES, REPORTED BY COUNTRIES OR AREAS OF DESTINATION, FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Countries or areas of destination	Crop seasons beginning September 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
Europe (European Economic Community)						
Belgium–Luxembourg	43†	55	68	65	62	41
France	170	239	228	261	279	173
West Germany	80	92	91	72	94	38
Netherlands	241	215	231	209	226	125
Totals	534	601	618	607	661	377
Other Europe						
Denmark	6	12	9	8	11	3
Finland	8	10	8	12	2	3
East Germany	..	..	..	..	27	4
Ireland	3	2	4	4	3	..
Norway	4	10	16	16	16	12
Sweden	22	47	54	69	53	48
Switzerland	10	3	4	3	2	1
United Kingdom	32	37	30	18	35	8
Other	3	3	4	2	3	..
Totals	88	124	129	132	152	79
Totals, Europe	622	725	747	739	813	456
Other countries or areas						
Canada	1,287	1,428	1,525	1,762	2,486	1,826
Mexico	4	5	2	5	2	7
Japan	11	23	18	28	32	34
West Indies	2	2	2	4	5	6
Other	13	10	10	9	5	6
Totals	1,317	1,468	1,557	1,808	2,530	1,879
Grand totals	1,939	2,193	2,304	2,547	3,343	2,335

*From USDA (1969*a*).
†Figures are stated as thousands of boxes of 80 pounds (about 36 kilograms) each.

equivalent to 24.4 per cent of the total fresh exports from the United States for that period. In this same crop season, Canada imported about 2.49 million boxes of fresh fruit which, when calculated from figures in table 8, amounted to 74.4 per cent of our total grapefruit exports. Therefore, Canada and the European countries, as shown in the table, have been the only substantial importers of United States grapefruit. Based on calculations from the 1966–67 production figures, the United States ordinarily exports only about 5.8 per cent of its total production as fresh grapefruit. Incidentally, at this writing, the chief users of fresh grapefruit exports to Europe are France, West Germany, and the Netherlands. From 1934 to 1967, exports of fresh grapefruit from the United States to the United Kingdom decreased from 264,000 to 8,000 boxes.

Grapefruit Products. In the early 1930's, a rapid expansion in grapefruit production occurred, with a concomitant rise in fruit-juice production and consumption, thus stimulating research into the development of processes for converting fresh fruit into high-grade juice products. The United States exports of grapefruit juices to general and specific areas from November 1, 1958, to October 31, 1968, are shown in table 9. Not only were Canada and Western Europe the chief importers of fresh grapefruit from the United States, they were also the chief importers of our grapefruit juices. From 1958–59 to 1967–68, however, total exports of single-strength grapefruit juice declined from approximately 5.4 million to 4.5 million gallons. During this same period, the total imports of single-strength grapefruit juice into Canada decreased from about 3.1 million to 2.3 million gallons. Similar decreasing trends were also noted for imports of single-strength grapefruit juice into some European countries.

The total exports of grapefruit segments have been on the wane since 1961–62. In the 1967–68 season, the total exports of canned grapefruit segments amounted to 92,000 cases (24 No. 2's), of which 62,000 cases, representing about 66 per cent of the total exports, were shipped to Canada (table 10).

World Exports and Imports (Fresh Fruit). The international export and import trade of fresh grapefruit is affected by the amount that is diverted to the processing plants for canning single-strength juice, concentrated juice, and segments, and for the making of special by-products. This effect, however, is at least partially offset by a substantial increase in world production of grapefruit. It should be noted that total production in the United States has materially decreased since the freezes of 1949 and 1951, which killed most of the bearing trees in Texas.

Rapid growth of the world output of grapefruit has caused serious thought about possible ways and means of marketing this expanded production. There is some indication that the European market is expanding and, if this trend prevails, a part of the increased production can be absorbed. The

TABLE 9

EXPORTS OF GRAPEFRUIT JUICE FROM THE UNITED STATES, REPORTED BY AREAS OF DESTINATION, FOR THE CROP SEASONS 1958–59 THROUGH 1967–68.*

Items and crop seasons	Canada	Europe					Other	Totals
		United Kingdom	Common Market†	Scandinavia‡	Other	Totals		
SINGLE-STRENGTH JUICE								
1958–59	3,104§	438	1,335	141	70	1,984	292	5,380
1959–60	3,305	255	749	160	102	1,266	156	4,727
1960–61	3,302	594	1,663	141	97	2,495	202	5,999
1961–62	3,266	960	2,527	199	168	3,854	240	7,360
1962–63	2,883	560	1,770	127	132	2,589	159	5,631
1963–64	1,855	128	490	53	71	742	160	2,757
1964–65	1,880	231	1,692	106	63	2,092	214	4,186
1965–66	2,028	29	668	90	59	846	235	3,109
1966–67	2,233	235	1,968	184	558	2,945	180	5,358
1967–68	2,344	6	1,476	195	247	1,924	204	4,472
CONCENTRATE: HOT PACK								
1958–59	10	..	24	16	27	67	112	189
1959–60	10	1	26	15	34	76	59	145
1960–61	42	..	49	16	18	83	147	272
1961–62	19	3	37	22	52	114	40	173
1962–63	6	2	56	22	28	108	24	138
1963–64	18	1	21	33	85	140	27	185
1964–65	48	2	46	26	34	108	36	192
1965–66	24	2	40	8	74	124	17	165
1966–67	11	1	27	15	81	124	18	153
1967–68	24	..	26	29	92	147	17	188
CONCENTRATE: FROZEN								
1958–59	135	..	5	..	2	7	19	161
1959–60	108	..	4	6	2	12	14	134
1960–61	139	2	9	6	..	17	37	193
1961–62	147	..	78	13	2	93	24	264
1962–63	164	..	4	3	1	8	21	193
1963–64	157	..	8	1	2	11	13	181
1964–65	155	2	6	..	3	11	35	201
1965–66	183	..	6	1	2	9	59	251
1966–67	207	3	9	2	3	17	60	284
1967–68	225	70	4	..	5	79	51	355

*From USDA (1969*b*, February).
†Belgium, France, Italy, Luxembourg, Netherlands, and West Germany.
‡Denmark, Norway, Sweden, and Finland.
§Figures are stated as thousands of gallons. (1,000 gallons equal about 3,785 liters.)

TABLE 10

EXPORTS OF CANNED GRAPEFRUIT SECTIONS FROM THE UNITED STATES, REPORTED BY COUNTRIES OF DESTINATION, FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Countries of destination	Crop seasons beginning November 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
Canada	55†	88	47	65	35	62
Europe						
Ireland	29	10	9	8	6	2
Netherlands	5	2	4	3	3	1
United Kingdom	106	28	28	33	20	4
Other	7	9	8	6	11	10
Totals	147	49	49	50	40	17
Other Countries						
Japan	2	2	2	3	2	4
Other	7	8	7	10	6	9
Totals	9	10	9	13	8	13
Grand totals	211	147	105	128	83	92

*From USDA (1969*a*).
†Figures are stated as 1,000 cases of 24/2's (30 pounds). Thirty pounds equal almost 14 kilograms.

United States can expect considerable competition in exports to Europe by other prominent grapefruit-producing areas.

Israel is not only the second largest grapefruit-producing country in the world, it is also the second largest exporter of the fresh fruit to world markets. The development of Israel's citrus industry is dependent upon the export market for absorbing a large proportion of its total output of fresh grapefruit, which was estimated to approach 7.6 million boxes in 1968–69. As shown in table 11, 1962 through 1968, the United Kingdom was the largest single market for Israel's exports, while West Germany, France, and Belgium-Luxembourg were, as listed, the next 3 most important markets for fresh grapefruit, with the Netherlands following closely. In 1967–68, Israel exported an estimated 4.3 million boxes of the fresh fruit to continental European countries and the United Kingdom. The latter country imported 1.07 million boxes, which was equivalent to 25 per cent of Israel's total exports. West Germany and France were also substantial consumers of Israel's crop, importing 1.2 million and 829,000 boxes, respectively.

The countries importing relatively small volumes of fresh grapefruit included Belgium-Luxembourg, Denmark, the Netherlands, Sweden, and Switzerland, but the imports into all these countries showed an increasing trend

TABLE 11

EXPORTS OF FRESH GRAPEFRUIT FROM ISRAEL, REPORTED BY COUNTRIES OF DESTINATION, FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Countries of destination	Crop seasons beginning October 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
Europe (European Economic Community)						
Belgium–Luxembourg	94†	134	158	192	232	288
France	274	372	447	538	728	829
West Germany	409	397	590	763	940	1,216
Italy	...	1	18	76	95	131
Netherlands	78	134	120	144	193	283
Totals	855	1,038	1,333	1,713	2,188	2,747
Other Europe						
Austria	20	24	26	37	45	47
Denmark	49	45	43	45	46	42
Finland	21	25	37	50	46	47
Norway	17	15	19	20	19	19
Romania	...	8	6	6	...	6
Sweden	49	57	61	64	66	68
Switzerland	144	160	133	131	185	194
United Kingdom	462	622	598	723	895	1,073
Other	16	12	17	13	16	17
Totals	778	968	940	1,089	1,318	1,513
Totals, Europe	1,633	2,006	2,273	2,802	3,506	4,260
Other countries	3	2	1	2	3	33
Grand totals	1,636	2,008	2,274	2,804	3,509	4,293

*From USDA (1969*a*).
†Figures are stated as thousands of actual boxes as reported from 1962–63 through 1965–66. Thereafter, they are converted to thousands of equivalent boxes of 80 pounds (about 36 kilograms).

between the crop seasons 1962–63 and 1967–68 (table 12). During this same period, the Netherlands was exceptional in that imports of fresh grapefruit were substantially increased from 248,000 to 504,000 boxes.

In 1967–68, the United Kingdom imported about 2.39 million boxes of fresh grapefruit from many countries of origin (table 13). In this crop season, the combined imports into the United Kingdom from Israel and South Africa were 1.7 million boxes, which was 71 per cent of Great Britain's total.

The West Indies and Cyprus annually export substantial amounts of fresh grapefruit to the British Isles. The United States exports of the fresh fruit

TABLE 12

TOTAL ANNUAL IMPORTS OF FRESH GRAPEFRUIT INTO SMALLER COUNTRIES OF WESTERN EUROPE FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Importing countries	Crop seasons beginning September 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
Belgium–Luxembourg	185†	229	257	273	308	317
Denmark	68	90	93	96	110	...
Netherlands	248	290	343	313	421	504
Sweden	89	112	123	128	129	115
Switzerland	196	223	208	215	269	239
Totals	786	944	1,024	1,025	1,237	1,175

*From USDA (1969*a*).
†Figures are stated as thousands of boxes of 80 pounds (about 36 kilograms) each.

TABLE 13

FRESH GRAPEFRUIT IMPORTED INTO THE UNITED KINGDOM, REPORTED BY COUNTRIES OR AREAS OF ORIGIN, FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Countries or areas of origin	Crop seasons beginning October 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
United States	28†	36	30	15	40	5
Europe						
Cyprus	220	266	386	315	367	396
Ireland	3	2	2	...	1	...
Netherlands	14	8	6	1	7	9
Portugal	...	2	...	...	...	...
Spain	22	25	22	21	31	40
Totals	259	303	416	337	406	445
Other countries or areas						
Argentina	5	6	9	2	1	1
Brazil	55	40	19	4	3	19
British Honduras	6	41	16	...	...	...
Cuba	10	...	...	...	...	...
Israel	473	668	654	767	863	1,030
Jamaica	56	68	38	55	39	42
Morocco	42	41	13	15	28	21
Mozambique	34	52	52	62	108	52
South Africa	432	606	588	621	653	668
Trinidad	115	91	64	118	54	34
Windward Islands	20	22	34	34	44	56
Other	31	55	32	20	49	20
Totals	1,279	1,690	1,519	1,698	1,842	1,943
Grand totals	1,566	2,029	1,965	2,050	2,288	2,393

*From USDA (1969*a*).
†Figures are stated as thousands of boxes of 80 pounds (about 36 kilograms) each.

TABLE 14

EXPORTS OF FRESH GRAPEFRUIT FROM SOUTH AFRICA, REPORTED BY COUNTRIES OF DESTINATION, FOR THE CALENDAR YEARS 1963 THROUGH 1968.*

Countries of destination	Calendar years					
	1963	1964	1965	1966	1967	1968
European (European Economic Community)						
Belgium–Luxembourg	13†	10	...	...	11	16
France	40	136	145	183	206	187
West Germany	34	122	150	311	305	394
Italy	...	...	...	...	30	41
Netherlands	15	6	...	5	...	3
Totals	102	274	295	499	552	641
Other Europe						
Denmark	2	3	3	7	4	5
Norway	5	...	5	7	5	6
Sweden	5	4	6	7	...	3
Switzerland	14	17	9	21	25	36
United Kingdom	440	586	613	747	672	849
Other	5	4	10	16	32	41
Totals	471	614	646	805	738	940
Totals, Europe	573	888	941	1,304	1,290	1,581
Other countries	3	8	16	15	19	10
Grand totals	576	896	957	1,319	1,309	1,591

*From USDA (1969a).
†Figures are stated as thousands of boxes of 80 pounds (about 36 kilograms) each.

into the United Kingdom have been on a steady decline for more than 30 years. From 1962–63 through 1967–68, the grand total of imports into the United Kingdom increased approximately from 1.6 to 2.4 million boxes.

South Africa has shown a continuous increase in fresh grapefruit exports, extending from a total value of 576,000 boxes in 1963 to about 1.6 million boxes in 1968, covering a 6-year period (table 14). In 1968, South Africa exported 849,000 boxes to the United Kingdom, which represented 53.4 per cent of its total exports to Western Europe. Within the calendar years 1963–68, South Africa increased its exports to the European Economic Community (EEC) from 102,000 to 641,000 boxes.

The summary data in table 15 show the total amount of fresh grapefruit imported into West Germany from the various large and small producing

TABLE 15

IMPORTS OF FRESH GRAPEFRUIT INTO WEST GERMANY, REPORTED BY COUNTRIES OR AREAS OF ORIGIN, FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Countries or areas of origin	Crop seasons beginning September 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
United States	164†	164	166	125	133	42
Europe						
Cyprus	23	62	77	130	194	267
Italy	12	11	14	5	3	6
Spain	37	33	45	39	32	40
Totals	72	106	136	174	229	313
Other countries or areas						
Algeria	26	20	26	13	6	13
Brazil	54	19	31	30	23	22
British Honduras	...	2	1	...	1	...
Honduras	23	74	82	117	70	68
Israel	445	430	610	780	873	1,177
Morocco	45	95	63	49	34	1
Mozambique	8	...	...	3	3	...
Paraguay	3	10	26	12	...	...
South Africa	54	96	171	238	319	253
Surinam	...	6	6	1	3	5
Turkey	15	12	19	13	11	3
U.A.R.	7	3	9	3	5	15
West Indies	36	24	17	22	46	7
Other	2	8	11	18	2	18
Totals	718	799	1,072	1,299	1,396	1,582
Grand totals	954	1,069	1,374	1,598	1,758	1,937

*From USDA (1969*a*).
†Figures are stated as thousands of boxes of 80 pounds (about 36 kilograms) each.

areas. It can readily be seen that for many years there has been a continued increase in imports into West Germany from Israel and Cyprus. In the 1967–68 crop season, the Mediterranean area (Algeria, Cyprus, Italy, Spain, Morocco, and Israel) exported a total of over 1.50 million boxes of fresh fruit to West Germany, and this amount represented 77.6 per cent of its total grapefruit imports. Besides being the first and second largest producers of grapefruit in the Mediterranean area, Israel and Cyprus were also the largest exporters to European markets. Their combined exports to West Germany amounted to nearly 1.45 million boxes, which was equivalent to about 95 per

TABLE 16

IMPORTS OF FRESH GRAPEFRUIT INTO FRANCE, REPORTED BY COUNTRIES OR AREAS OF ORIGIN, FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Countries or areas of origin	Crop seasons beginning October 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
United States	153†	245	202	246	260	159
Europe						
Cyprus	23	21	17	28	37	14
Italy	...	...	...	...	1	...
Spain	2	6	11	11	18	11
Totals	25	27	28	39	56	25
Other countries or areas						
Algeria	49	57	41	39	27	25
Israel	291	413	479	589	688	907
Morocco	57	37	52	22	45	52
Mozambique	17	2	...	29	...	...
South Africa	58	114	153	127	181	136
Surinam	5	8	7	7	10	13
Tunisia	7	7	7	7	5	3
Other	16	25	19	20	29	59
Totals	500	663	758	840	985	1,195
Grand totals	678	935	988	1,125	1,301	1,379

*From USDA (1969*a*).
†Figures are stated as thousands of boxes of 80 pounds (about 36 kilograms) each.

cent of the total exported to that country from the Mediterranean area. South Africa has been by far the major exporter of fresh grapefruit in the Southern Hemisphere. The grand total imported into West Germany from all sources in 1967–68 amounted to almost 1.94 million boxes, which was the highest quantity reported, beginning with the 1962–63 crop season.

The total imports of fresh grapefruit into France have continued to increase since 1962–63. For the 6 seasons indicated in table 16, France imported its major proportion of the fresh fruit from Israel, and in 1967–68, the volume imported from Israel amounted to 907,000 boxes, which was equivalent to 65.7 per cent of the total of such imports into France. The United States and South Africa have been the second and third largest exporters to France.

The data in table 17 show the importation of fresh grapefruit into various Western European countries, including the United Kingdom, from specified countries for 1966 through 1968, broken down into calendar years. The

TABLE 17

IMPORTS OF FRESH GRAPEFRUIT INTO WESTERN EUROPE, REPORTED BY SPECIFIED COUNTRIES OR AREAS, IN THE YEARS 1966 THROUGH 1968.*

Importing countries	Countries or areas of origin							
	Cyprus	Honduras	Israel	Morocco	South Africa	United States	Other	Totals
EEC:								
Belgium								
1966	...†	...	194	...	...	65	12	271
1967	...	...	204	...	...	77	30	311
1968	...	...	244	...	...	...	89	333
France								
1966	28‡	15	585	35	125	251	94	1,133
1967	24	10	652	65	185	278	85	1,299
1968	23	25	1,029	25	132	134	119	1,487
West Germany								
1966	145	105	822	55	269	128	149	1,673
1967	210	68	860	23	313	109	147	1,730
1968	265	67	1,315	1	245	21	137	2,051
Italy								
1966	...	...	80	...	...	...	14	94
1967	...	...	91	...	35	...	9	135
1968	...	...	150	...	...	...	32	182
Netherlands								
1966	5	46	196	...	6	130	148	531
1967	9	7	184	5	3	92	132	432
1968	18	36	314	...	2	67	143	580
Totals, EEC								
1966	178	166	1,877	90	400	574	417	3,702
1967	243	85	1,991	93	536	556	403	3,907
1968	306	128	3,052	26	379	222	520	4,633
Other Europe								
Austria								
1966	...	...	41	★	6	9	2	58
1967	1	★	52	★	14	4	5	76
1968	3	...	61	...	10	1	7	82
Denmark								
1966	7	17	47	★	7	14	9	101
1967	10	12	52	1	6	14	6	101
1968	11	22	43	★	6	3	7	92
Finland								
1966	1	1	46	...	7	5	1	61
1967	★	★	46	...	9	4	1	60
1968	4	2	44	...	10	2	3	65
Norway								
1966	3	...	20	...	7	13	1	44
1967	2	★	17	...	6	13	1	39
1968	2	...	19	...	6	8	2	37
Sweden								
1966	5	★	63	...	6	44	7	125
1967	1	1	70	...	3	46	6	127
1968	1	1	68	...	8	26	9	113
Switzerland								
1966	18	★	141	...	18	30	8	215
1967	17	...	197	...	24	31	22	291
1968	29	...	186	...	23	8	7	253
United Kingdom								
1966	318	...	825	26	477	15	450	2,111
1967	408	...	773	29	487	40	497	2,234
1968	473	...	1,152	13	525	5	392	2,560
Grand totals								
1966	530	184	3,060	116	928	704	894	6,417
1967	682	98	3,198	123	1,085	708	941	6,835
1968	829	153	4,625	39	967	275	947	7,835

*From USDA (1969*b*, December).
†Blanks mean "data not available."
‡Figures are stated as thousands of boxes of 80 pounds (about 36 kilograms) each.
★Stars mean "fewer than 500 boxes."

United States exports to Europe are shown to have declined substantially, decreasing from 708,000 to 275,000 boxes during the 2-year period, 1967–68. Imports into various markets continued to increase in 1968 to a total of 7.8 million boxes, about 15 per cent above the grand total for 1967. The United Kingdom, West Germany, and France increased imports to a total of about 6.1 million boxes, which amounted to over 15 per cent above their combined totals shown for 1967. Israel not only increased its total production of grapefruit, but also expanded exports to 4.3 million boxes, which was equivalent to 55 per cent of the total imports in 1968—more than double the volume supplied in 1963–64 (see table 11). Cyprus doubled its grapefruit production in 5 years, as well as its exports, reaching a value of 829,000 boxes for 1968. Imports from South Africa and the United States declined significantly in 1968. The EEC imported 4.6 million boxes in 1968, which was equivalent to 60 per cent of the total imports into Western Europe.

Imports of Grapefruit Juice Products by the United Kingdom. In accordance with the upward trend in world production of grapefruit, citrus producers everywhere are developing facilities for converting the fresh fruit into processed forms. Such facilities are well established in Israel, Spain, and Italy, as well as in the Caribbean area, Latin America, and South Africa. The processing of grapefruit into various juice products is certainly an excellent expedient for using surplus fresh fruit in a different form, as well as for developing diversified commodities for an expanding market.

The origins of imports of grapefruit juices and segments by the United Kingdom are indicative of the extent of developments of facilities for processing the fruit in countries other than the United States. The data recorded in table 18 show that Israel and Trinidad-Tobago were the principal exporters of single-strength sweetened and unsweetened grapefruit juice to the United Kingdom, and that in 1967–68, exports from these 2 areas represented 83.5 per cent of England's total imports of these juice products.

Great Britain imports considerable quantities of single-strength sweetened juice from several producing areas; chief among these are Israel, Jamaica, and Trinidad-Tobago. Total imports for this particular type of juice in 1963–64 averaged approximately 2.6 million gallons, which was equal to over 85 per cent of the total single-strength juice imported during that period. At this time, imports from the United States were on a continuous decline. Imports of single-strength sweetened grapefruit juice into the United Kingdom from Israel were on the increase, and in 1963–64, Israel's exports (1.333 million gallons) surpassed those of Trinidad-Tobago (987,000 gallons). The United Kingdom used relatively small quantities of concentrated unsweetened grapefruit juice (the mean for 3 years was 132,000 gallons). Again, the chief exporters of this type of juice to Great Britain were Israel, Jamaica, and South Africa.

TABLE 18

IMPORTS OF GRAPEFRUIT JUICES AND OTHER PRODUCTS INTO THE UNITED KINGDOM, REPORTED BY COUNTRIES OR AREAS OF ORIGIN, FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Items and countries or areas of origin	Crop seasons beginning November 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
Single-strength juice†						
United States	546‡	107	240	26	224	7
British Honduras	90	135	79	119	273	106
Israel	581	1,333	1,942	1,616	1,992	2,779
Jamaica	364	284	458	422	287	323
South Africa	51	195	116	142	97	180
Trinidad–Tobago	1,172	987	1,313	1,183	706	590
Other	36	9	13	25	39	48
Totals	2,840	3,050	4,161	3,533	3,618	4,033
Concentrated unsweetened juice						
British Honduras	4	9	19	2	11	4
Israel	17	43	39	29	52	156
Jamaica	72	46	67	79	49	46
South Africa	17	36	41	72	7	6
Other	5	3	9	15	52	55
Totals	115	137	175	197	171	267
Grapefruit, tinned or bottled, in syrup						
United States	186§	22	27	29	19	7
British Honduras	117	135	67	180	218	220
Cyprus	53	62	42	51	97	84
Israel	578	507	485	626	730	886
Jamaica	457	564	633	578	504	539
South Africa	145	266	199	320	333	454
Trinidad–Tobago	127	97	93	138	180	111
Other	29	18	5	12	48	60
Totals	1,692	1,671	1,551	1,934	2,129	2,361

*From USDA (1969*a*).
†Includes both sweetened and unsweetened juices.
‡Figures are stated as thousands of gallons (1,000 gallons equal about 3,785 liters).
§Figures are stated as thousands of cases (equivalent) of 24 number 2 cans.

TABLE 19

IMPORTS OF CANNED GRAPEFRUIT SEGMENTS INTO THE UNITED KINGDOM, REPORTED BY COUNTRIES OR AREAS OF ORIGIN, FOR THE CROP SEASONS 1962–63 THROUGH 1967–68.*

Countries or areas of origin	Crop seasons beginning October 1					
	1962–63	1963–64	1964–65	1965–66	1966–67	1967–68
United States	241†	23	27	30	18	4
West Indies						
British Honduras	141	135	67	180	218	219
Jamaica	466	562	626	574	517	530
Trinidad–Tobago	125	87	101	136	184	107
Totals	732	784	794	890	919	856
Other countries or areas						
Cyprus	52	55	48	53	94	88
Israel	571	483	477	651	714	876
South Africa	166	255	217	306	370	464
Other	30	20	4	8	21	16
Totals	819	813	746	1,018	1,199	1,444
Grand totals	1,792	1,620	1,567	1,938	2,136	2,304

*From USDA (1969*a*).
†Figures are stated as thousands of cases (equivalent) of 24 number 2 cans.

The imports of concentrated unsweetened juice have shown an upward trend since 1962–63, increasing to 267,000 gallons in 1967–68. Israel and Jamaica continued to be the first and second most important suppliers in 1967–68. The United Kingdom also imported large volumes of grapefruit, tinned or bottled, in syrup, the amounts reaching 2.4 million cases (24 No. 2's) in 1967–68. The 3 prominent suppliers of this product were Israel, Jamaica, and South Africa. In 1967–68, their combined exports to the United Kingdom amounted to 79.6 per cent of the total imports (table 18).

The consumption of canned segments in the United Kingdom has been on an upward trend since 1962–63 (table 19). The grand total of imports increased from 1.8 million cases in 1962–63 to about 2.3 million cases in 1967–68. The United Kingdom imported more canned grapefruit segments than any other country during the 6-year period, 1962–63 through 1967–68. During this interval, the imports of canned grapefruit segments from the United States decreased to relative insignificance. In 1967–68, the West Indies exported 856,000 cases (24 No. 2's) to the United Kingdom, and this amount was equivalent to 37.1 per cent of the total canned segments imported into

that area. For the same period, the combined imports of canned grapefruit segments into the United Kingdom from both Israel and South Africa amounted to 1.34 million cases, which was equivalent to 58.1 per cent of the total imports. The sum of the imports from the West Indies, Israel, and South Africa was 95.2 per cent of the total supplies of canned grapefruit segments imported into the United Kingdom. Approximately 90 per cent of the grapefruit segments produced by Israel and most of the canned segments produced in the West Indies (British Honduras, Jamaica, and Trinidad-Tobago) are destined for the United Kingdom.

Frozen Citrus Concentrate in Relation to Other Juice Products. The phenomenal growth in the production of frozen citrus concentrates indicates that these products are not only highly acceptable to the public, but that they are also capable of competing with both fresh citrus fruits and other types of juice products, such as canned single-strength juice and pasteurized concentrate. Frozen orange concentrate, the first to be developed from citrus, was introduced into Florida in 1945. It was the first processed juice that possessed the qualities of flavor and aroma that were comparable to freshly extracted juice, and it was the first product that could successfully substitute for fresh juice. Its commercial development was actually born of the necessity to merchandise increasingly larger crops year after year.

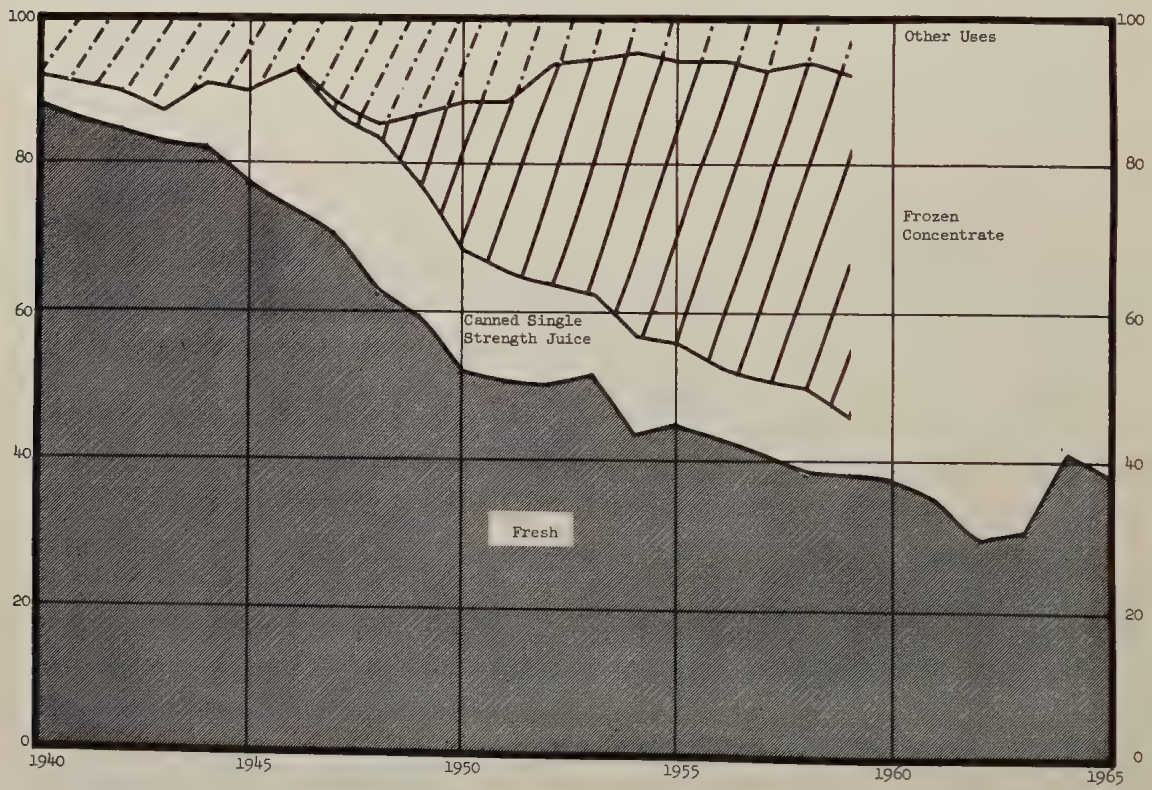


Fig. 8. Changes in the diversion and utilization of the total United States orange and tangerine crops, 1940 through 1965. Data are expressed in percentages, as fresh-fruit equivalents. (From Sunkist Growers, Inc., 1967.)

Frozen orange concentrate is used here as an example to show the effect this product has had on the diversion and utilization of the total orange and tangerine crop (figure 8). This figure shows that in 1940 approximately 88 per cent of the crop was utilized as fresh fruit and 4.2 per cent as canned single-strength juice, while the remaining percentage was assigned to other uses. Since 1942–43, orange processing in Florida has increased at a rate nearly parallel with orange production, but the proportion of the crop that has been utilized as fresh fruit has been on a steady decline since 1940. In 1957–58, 40.7 per cent of the total production of oranges and tangerines in the United States was estimated to be the amount of fruit (fresh-fruit equivalent) used for producing frozen concentrate.

In 1968–69, Florida produced 129.7 million boxes of oranges, of which 92.1 million boxes (71 per cent of the total crop) were used to produce 103.8 million gallons of frozen concentrate (table 20). This extraordinary increase in frozen concentrate occurred at the expense of both the use of fresh oranges and canned single-strength orange juice (figure 8). This trend of using increasingly more of the crop for processing into canned and frozen products has also characterized other commercial varieties of citrus, such as grapefruit and lemons. In fact, frozen orange concentrate, as a result of its high quality, can compete successfully with frozen grapefruit or lemon concentrate at the retail level. In the sale of processed citrus juices, some consumers vary their purchases in response to changes in the relative prices of the different juice products.

Table 20 shows the annual packs of both frozen and nonfrozen concentrates for Florida oranges and grapefruit. In the 1968–69 crop season, Florida produced 5.9 million gallons of frozen grapefruit concentrate, which amounted to only 5.7 per cent of the total production of frozen orange concentrate. Processed grapefruit concentrate or nonfrozen concentrate has been produced in Florida in relatively small quantities—55,000 gallons in 1964–65; in California, the production amounted to 277,000 gallons in 1958–59, the last year data were reported (table 21). As indicated by the absence of data, no frozen grapefruit juice in either single-strength or concentrated forms had been produced in the California–Arizona citrus area. Florida was the only citrus-processing area in the United States that produced frozen concentrated blended juices, ranging in variable amounts from 965,000 gallons in 1953–54 to 36,000 gallons in the 1968–69 crop year (table 20).

After the introduction of canned single-strength orange juice into Florida in 1929, the market for this product expanded greatly, but single-strength orange juice never possessed the quality of either freshly extracted juice or whole fresh fruit, and consequently it never became a close competitor with either of these items. According to Shear and Joslyn (1954), the national hot pack of single-strength citrus juices between 1947 and 1951 amounted, on the

TABLE 20

FLORIDA CITRUS PRODUCTS: ANNUAL PACKS OF FROZEN AND PROCESSED CONCENTRATES, FRESH ORANGE JUICE, CITRUS FEED, AND CITRUS MOLASSES FOR THE CROP SEASONS 1947–48 THROUGH 1968–69.*

Crop seasons	Concentrated orange juice				Chilled orange juice		Concentrated grapefruit juice		Blended juice	Citrus feed (1,000 tons)	Citrus molasses (1,000 tons)
	Frozen		Processed								
	Frozen product (1,000 gallons)	Fruit used (1,000 boxes)	Finished product (1,000 gallons)	Fruit used (1,000 boxes)	Finished product† (1,000 gallons)	Fruit used (1,000 boxes)	Frozen finished product (1,000 gallons)	Processed finished product (1,000 gallons)	Frozen finished product (1,000 gallons)		
1947–48	1,936	1,600	1,739	3,316	.....‡	.....	1	1,440	....	154	66
1948–49	10,233	8,320	1,898	2,500	.....	.....	116	19	112	134	41
1949–50	21,647	17,797	1,529	1,851	.....	.....	1,585	28	1,303	163	41
1950–51	30,758	23,084	2,530	2,770	.....	.....	188	148	245	188	70
1951–52	44,031	31,579	1,898	1,468	.....	.....	1,098	16	536	218	54
1952–53	46,554	32,646	537	399	.....	.....	1,226	51	480	223	39
1953–54	65,531	48,272	1,339	1,101	.....	.....	1,656	55	965	288	53
1954–55	64,686	44,643	1,531	1,120	16,686	3,001	1,155	32	561	262	49
1955–56	70,224	49,119	1,086	743	20,103	3,484	2,512	31	954	297	42
1956–57	72,012	48,741	1,801	1,163	32,040	5,619	2,949	59	597	297	60
1957–58	57,151	43,810	1,149	770	32,789	6,044	3,330	108	507	292	36
1958–59	79,911	52,437	547	319	34,506	6,129	4,952	188	690	321	44
1959–60	78,149	51,748	378	112	40,932	7,089	1,613	27	284	284	29
1960–61	84,298	55,819	155	111	32,825	5,679	3,841	20	256	320	33
1961–62	116,082	73,716	278	158	41,763	7,298	3,163	116	267	420	55
1962–63	51,648	47,121	55	54	27,251	5,550	2,323	36	53	311	31
1963–64	53,674	34,176	41	30	28,058	4,891	2,573	21	264	195	26
1964–65	88,869	54,487	70	24	41,857	7,300	4,000	55	70	334	49
1965–66	76,695	61,805	49	28	67,643	12,325	3,971	31	50	422	56
1966–67	131,755	96,752	119	94	93,348	16,491	5,485	32	29	574	99
1967–68	83,697	61,965	22	17	90,013	16,016	1,814	...	10	420	52
1968–69	103,750	92,125	31	29	88,565	17,855	5,920	...	36	601	78

*From Florida Department of Agriculture (1969).
†The production for 1954–55 through 1959–60 is estimated.
‡Blanks mean "data not available."
Note: The metric conversions for quantities shown below headings are: 1,000 gallons equal about 3,785 liters; standard boxes hold 80 pounds (about 36 kilograms); 1 short ton of 2,000 pounds equals 908 kilograms.

TABLE 21

CALIFORNIA AND ARIZONA CITRUS PRODUCTS: ANNUAL PRODUCTION* OF FROZEN CITRUS JUICE, PROCESSED CONCENTRATE, AND FRESH CITRUS JUICE IN THE CROP SEASONS 1947-48 THROUGH 1964-65.†

Crop seasons	Frozen orange juice		Frozen grapefruit juice		Other concentrated juice		Fresh juice	
	Single-strength	Concentrate	Single-strength	Concentrate	Orange	Grapefruit	Orange	Grapefruit
1947–48	832‡	437	2	..	2,928	227	2,764	24
1948–49	528	1,963	..	..	2,769	132	2,685	56
1949–50	432	3,490	..	80	3,216	304	2,314	23
1950–51	202	4,180	4	..	3,251	196	2,397	15
1951–52	264	3,712	..	..	1,654	128	3,028	23
1952–53	157	4,710	..	..	3,451	220	2,545	12
1953–54	39	1,447	..	21	2,346	264	1,150	9
1954–55	382	3,872	..	..	2,737	225	4,337	8
1955–56	...§	3,837	..	30	3,534	277	3,066	..
1956–57	495	3,055	..	..	3,808	256	5,236	..
1957–58	288	1,480	..	..	2,102	247	4,148	..
1958–59	...	3,688	..	46	3,729	277	6,175	..
1959–60	...	2,900	..	..	.....	...	.....	..
1960–61	...	2,400	..	..	.....	...	.....	..
1961–62	...	.....	..	..	.....	...	.....	..
1962–63	...	.....	..	..	.....	...	.....	..
1963–64	...	.....	..	..	.....	...	.....	..
1964–65	...	.....	..	..	.....	...	.....	..

*Excluding lemon products.
†From Florida Department of Agriculture (1965).
‡Figures are stated as thousands of gallons (1,000 gallons equal about 3,785 liters).
§Blanks mean "data not available."

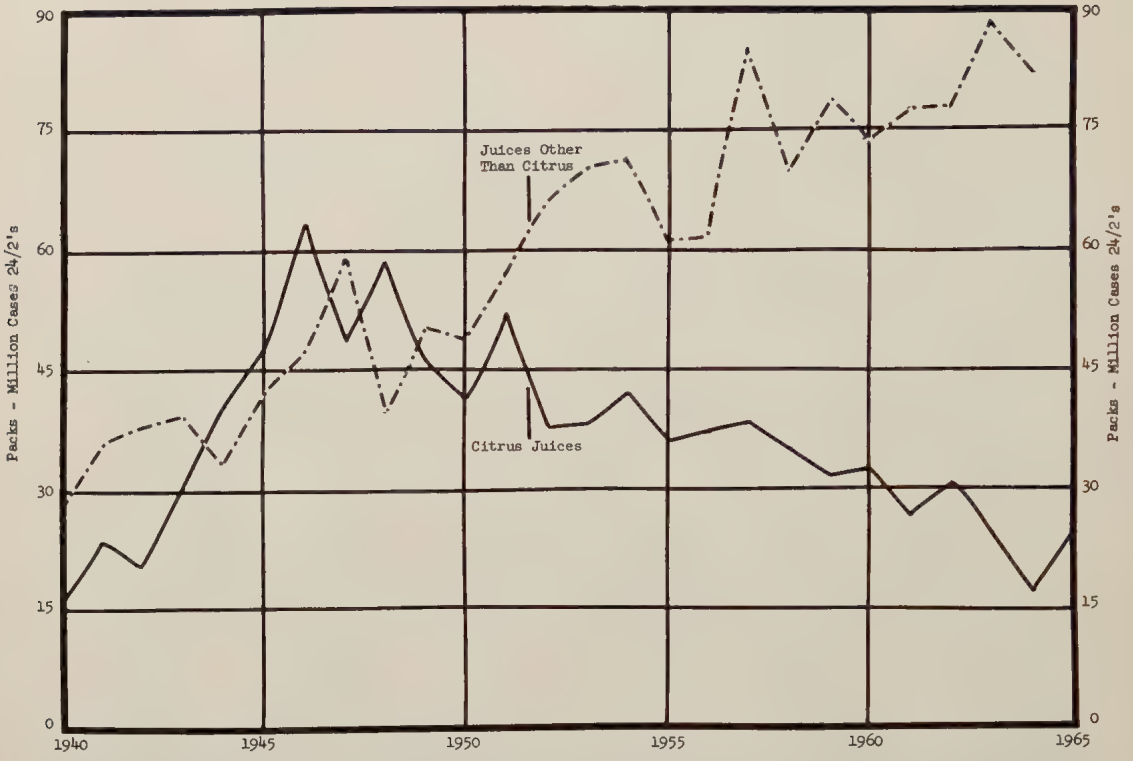


Fig. 9. Utilization of citrus juices and other fruit juices, 1940 through 1965. The noncitrus juices included canned single-strength fruit and tomato juice as well as United States imports of pineapple juice. (From Sunkist Growers, Inc., 1967.)

average, to 47 million cases, of which orange juice accounted for 47 per cent; grapefruit, 30 per cent; blended juices, 20 per cent; tangerines, 2 per cent; and lemon juice, approximately 1 per cent. During and after World War II, Florida's production of single-strength orange juice reached a peak of 27 million cases in 1947. Since then, the pack has been steadily but slowly declining as a result of a plentiful supply of hot packs of other fruit juices and the continued acceptance by consumers of frozen concentrated orange juice (figures 8 and 9).

As a result of the increased production of grapefruit, canned hot-packed grapefruit juice was initiated on a small scale in Florida in 1926. This juice industry developed very slowly at first, but with continued expansion in production, and with the simultaneous improvement in the juice pack, canned single-strength juice became a major outlet for grapefruit before World War II. With further increase in production plus market demands created by the war, canned hot-packed single-strength grapefruit juice continued to increase more rapidly than orange juice until a peak of 26 million cases was packed in 1943 and also in 1945. The average annual pack of grapefruit juice has continued to be much less than that of the 2 peak years, while the output of orange juice has continued to increase and exceed that of grapefruit.

Frozen grapefruit concentrate was first produced in Florida in 1948, and the pack increased to nearly 1 million gallons during 1949–51, but subsequently the annual production rate has varied greatly, and at no time has the rate of growth approached that of frozen orange concentrate. In the United States, Florida produces more than 70 per cent of all fresh grapefruit shipments and more than 90 per cent of the tonnage used by processors. Florida processes more than 50 per cent of its grapefruit crop, which is slightly more than the average for the national totals. Approximately 80 per cent of the processed tonnage has been converted to juice and 20 per cent to canned segments. With the introduction of frozen citrus concentrate, the production of pasteurized single-strength grapefruit and orange juices has been on a steady decline, while the frozen concentrate pack has increased steadily.

The utilization of single-strength citrus juices reached its peak in the 1945–46 crop season, and slowly declined until 1965, when studies ended, while juices made from other fruits continued to increase (figure 9). The decline in the hot pack of single-strength citrus juices after 1945 was significantly correlated with the enormous increase in production and sale of frozen concentrated orange juice. Over a 5-year period, 1959–60 through 1963–64, the average pack of single-strength citrus juice amounted to 26.3 million cases (24 No. 2 cans), which was equivalent to 24.7 per cent of the total fruit- and tomato-juice pack. During this same period, the average annual fruit-juice pack amounted to 72.1 million cases, and the average annual tomato-juice pack amounted to 34.1 million cases, the sum of which brought the total average annual fruit- and tomato-juice packs to more than 106 million cases (table 22).

Cost of Producing and Marketing Grapefruit. The cost of producing and marketing a box of grapefruit varies considerably from year to year, and is influenced by yields as well as costs of labor, equipment, and materials, such as fertilizers, sprays, and dust for pest control. The costs also vary in different states according to natural conditions and distances from the markets. Production and marketing costs are consistently higher in California than in Florida. There are several inherent conditions to cause this: irrigation water costs, wage scales, taxes and, again, distances from the markets.

In his studies on the production costs and returns for 193 groves in Florida during a period of 33 years (1931–64), Savage (1966) listed the total operating costs under 5 items: (1) labor, power, and equipment; (2) fertilizer materials; (3) spray and dust materials; (4) state and county taxes; and (5) miscellaneous costs. The distribution of the 33-year averages of the total costs among these items is shown in table 23. For groves over 10 years of age, labor, power, and equipment required, on the average, 46.3 per cent of the total operating costs, and the fertilizer materials accounted for 32.1 per cent; spray and dust ma-

TABLE 22

United States Packs of Fruits in Various Forms and United States Imports of Pineapple Juice in the Crop Seasons 1939–40 through 1967–68.*

Crop seasons	Canned single-strength fruit and tomato juices								Frozen fruit juices (b) (million pounds)	Canned fruits (c) (billion pounds)	Frozen fruits (b) (million pounds)	Dried fruits (d) (billion pounds)
	Citrus		Imports of pineapple juice (a) (million cases)	Grape (million cases)	Fruit nectars; apple, prune, and other juices (million cases)	Total fruit juices (million cases)	Tomato (million cases)	Total fruit and tomato juices (million cases)				
	Pack (million cases)†	Proportion of total fruit and tomato juices (per cent)										
1939–40	16.0	36.0	10.4	3.1	1.2	30.7	13.7	44.4	....	1.9	141	1.1
1940–41	23.8	40.2	14.4	3.2	2.7	44.1	15.1	59.2	....	1.8	173	0.8
1941–42	20.1	34.5	7.9	2.6	4.2	34.8	23.4	58.2	....	2.3	208	.9
1942–43	30.1	43.5	6.9	2.9	4.1	44.0	25.2	69.2	1	2.2	195	1.0
1943–44	41.0	55.4	4.4	1.6	4.2	51.2	22.8	74.0	6	1.6	187	1.3
1944–45	47.6	52.6	4.3	2.0	5.8	59.7	30.8	90.5	14	2.1	324	1.1
1945–46	62.9	57.1	10.5	2.6	5.7	81.7	28.4	110.1	20	1.9	427	1.0
1946–47	49.3	45.5	11.5	3.4	9.2	73.4	34.9	108.3	11	3.1	519	1.0
1947–48	58.4	59.7	9.9	3.5	6.6	78.4	19.5	97.9	10	2.6	344	1.1
1948–49	46.2	47.9	11.5	3.3	7.6	68.6	27.8	96.4	35	2.5	370	.9
1949–50	41.4	45.6	12.0	2.9	10.9	67.2	23.5	90.7	134	2.7	354	1.0
1950–51	51.8	47.5	13.7	3.0	14.3	82.8	26.2	109.0	313	2.7	472	.7
1951–52	38.1	36.6	12.2	4.8	12.8	67.9	36.2	104.1	386	3.1	416	.9
1952–53	38.1	35.2	16.9	4.2	13.2	72.4	35.8	108.2	588	2.8	420	.9
1953–54	42.0	37.1	16.4	4.2	12.7	75.3	37.8	113.1	712	2.9	542	.8
1954–55	36.1	36.9	16.0	4.5	14.1	70.7	27.0	97.7	757	3.0	523	.7
1955–56	37.0	37.6	16.2	5.0	13.4	71.6	26.9	98.5	839	3.5	660	.8
1956–57	38.5	31.2	16.7	5.1	19.6	79.9	43.6	123.5	898	3.6	694	.8
1957–58	35.0	33.4	12.5	3.8	20.8	72.1	32.6	104.7	938	3.4	671	.7
[illegible]	31.6	28.8	15.2	5.4	20.0	72.2	37.5	109.7	750	3.3	610	.6

1960–61	26.7	25.6	14.4	8.2	22.0	71.3	33.0	104.3	960	4.3	660	.7
1961–62	30.4	28.1	15.3	8.1	22.7	76.5	31.6	108.1	1,036	4.7	705	.8
1962–63	24.7	21.9	15.3	9.3	23.4	72.7	40.2	112.9	1.369	4.9	668	.7
1963–64	17.3	17.4	14.8	8.3	24.6	65.0	34.5	99.5	614	4.4	619	.8
1964–65	24.4	....‡	13.8	....	....	....	35.3	....	....	....	795	.8
1965–66	29.0	....	15.4	....	....	....	32.8	....	....	....	653	.9
1966–67	41.2	....	15.0	....	....	....	31.9	....	....	....	664	.8
1967–68	29.6	....	15.1	....	....	....	35.1	....	....	....	642	.7
Five-Year Averages												
39–40 through 43–44	26.2	41.9	8.8	2.7	3.3	41.0	20.0	61.0	4	2.0	181	1.0
44–45 through 48–49	52.9	52.6	9.5	3.0	7.0	72.4	28.3	100.6	18	2.4	397	1.0
49–50 through 53–54	42.3	40.4	14.2	3.8	12.8	73.1	31.9	105.0	427	2.8	441	.9
54–55 through 58–59	35.6	33.6	15.3	4.8	17.6	73.3	33.5	106.8	836	3.4	632	.7
59–60 through 63–64	26.3	24.7	14.6	8.3	22.8	72.1	34.1	106.1	1,013	4.5	654	.8

*From Sunkist Growers, Inc. (1967, 1969).
†Regarding the various amounts given below heads: One case consists of 24 number 2 cans, weighing 30 pounds (almost 14 kilograms). One pound equals 0.454 kilogram.
‡Blanks mean "data not available."
(a) Beginning 1947–48, figures represent packs including Hawaiian and foreign operations. Earlier seasons represent United States imports of pineapple juice.
(b) Processed weights.
(c) Canned weights. Includes apples, applesauce, peaches, pears, apricots, berries, cherries (also brined cherries), grapefruit segments, pineapple, figs, olives, cranberry sauce, fruits for salad and cocktail, plums, and prunes.
(d) Processed weights. Includes prunes, raisins, apples, apricots, dates, figs, peaches, and pears.

TABLE 23

PERCENTAGE COST OF EACH ITEM OF TOTAL OPERATING COSTS PER ACRE, 1931–64 AND 1963–64.*

Items of cost	Ages of groves in years			
	1931–64		1963–64	
	10 and under	Over 10	10 and under	Over 10
Labor, power, and equipment	52.0	46.3	60.4	52.7
Fertilizer materials	30.0	32.1	22.7	21.9
Spray and dust materials	6.8	10.1	8.5	13.6
State and county taxes	5.1	6.8	5.1	8.2
Miscellaneous	6.1	4.7	3.3	3.6
Total operating costs	100.0	100.0	100.0	100.0

*From Savage (1966).

TABLE 24

COSTS PER BOX FOR HAULING AND PICKING FLORIDA GRAPEFRUIT, AS REPORTED BY SELECTED FIRMS FOR THE CROP SEASONS 1953–54 THROUGH 1963–64.*
(Shown in cents and fractions of cents.)

Crop seasons	Number of firms reporting	Average hauling costs	Average picking costs	Combined average costs for hauling and picking
1953–54	37	9.61	20.58	30.19
1954–55	36	9.38	20.91	30.29
1955–56	36	9.47	21.73	31.20
1956–57	34	9.27	23.46	32.73
1957–58	34	11.31	24.09	35.40
1958–59	32	11.46	24.16	35.62
1959–60	33	11.23	25.16	36.39
1960–61	37	11.17	26.69	37.86
1961–62	33	10.41	25.75	36.16
1962–63	32	12.94	28.32	41.26
1963–64	30	13.73	31.47	45.20

*From Florida Department of Agriculture (1965).

terials amounted to 10.1 per cent. The combined taxes and miscellaneous costs varied, ranging from about 8 to 12 per cent. The costs of labor, power, and equipment exceeded the cost of fertilizer materials in 25 of the 33 seasons. The difference between the costs of these items increased during recent years, with the cost of labor, power, and equipment increasing faster than those of fertilizer materials. These cost differentials were further exemplified by the fact that 10 seasons (1932–42) occurred when the total operating costs did not equal the costs of the 1 item of labor, power, and equipment for any

one of the 18 seasons between 1946 and 1964. If the itemized costs of production, in percentages, for the groves over 10 years of age were to be converted to dollar values, the average operating costs per acre per season, when distributed into the 5 cost categories, would amount to $64.24 for labor, power, and equipment; $44.44 for fertilizer materials; $14.07 for spray and dust materials; $9.38 for state and county taxes; and $6.52 for miscellaneous costs. The sum of the itemized costs amounted to total operating costs of $138.65, which was equivalent to an average operating cost of 54 cents per box.

The cost of grapefruit production is usually reported as cost per box. This is obtained by dividing the total cultural cost per acre, exclusive of interest on investment and depreciation on trees, by the total yield per acre. In California, however, in those years where returns from by-products fruit are only sufficient to cover harvesting and handling and little or no cultural cost, the cultural costs of producing the entire crop must be borne by fresh fruit alone. Again, if the on-tree farm value (in dollars) is divided by the bearing acreage, the quotient obtained is the on-tree farm value per acre. Therefore, the difference between the on-tree farm value and cultural cost per acre is the income per acre, excluding interest on investment and depreciation on trees. With reference to the fresh-fruit market, the difference between the FOB and the on-tree prices of grapefruit is the cost of picking, hauling, packing, selling, and advertising. Since 1954, all costs have increased substantially.

TABLE 25

AVERAGE COSTS PER STANDARD BOX (EQUIVALENT) FOR PACKING AND SELLING FLORIDA GRAPEFRUIT FOR FRESH CONSUMPTION IN THE CROP SEASONS 1953–54 THROUGH 1963–64.*

(Shown in dollars and cents.)

Crop seasons	Containers used†	
	0.8-bushel cartons	1.6-bushel boxes
1953–54	0.9058	0.8375
1954–55	.9277	.7984
1955–56	1.0000	.8682
1956–57	1.0549	.9285
1957–58	1.1057	1.0077
1958–59	1.1305	.9719
1959–60	1.1804	.9462
1960–61	1.2042	1.0010
1961–62	1.2819	.9584
1962–63	1.2857	1.1581
1963–64	1.2857	1.0853

*From Florida Department of Agriculture (1965).
†One standard box contains 80 pounds (about 36 kilograms). One bushel equals about 35.24 liters.

TABLE 26

COSTS OF PRODUCING AND MARKETING CALIFORNIA–ARIZONA AND FLORIDA FRESH GRAPEFRUIT IN THE CROP YEARS 1939-40 THROUGH 1967–68.*
(Shown in dollars and cents per packed equivalent carton.)

Crop year (November–October)†	California summer					California–Arizona desert					Florida		
	Cultural costs‡	Picking and hauling	Packing	Selling and advertising	Total costs FOB packing house	Cultural costs	Picking and hauling	Packing	Selling and advertising	Total costs FOB packing house	Cultural costs	Picking, hauling, packing, and selling	Total costs FOB packing house
1939–40	0.21	0.05	0.22	0.04	0.53	0.11	0.05	0.23	0.04	0.43	0.20	0.41	0.61
1940–41	.23	.06	.22	.03	.54	.13	.06	.24	.03	.46	.18	.37	.55
1941–42	.25	.08	.30	.03	.66	.12	.06	.24	.03	.45	.20	.40	.60
1942–43	.30	.10	.26	.04	.70	.15	.09	.29	.04	.57	.18	.44	.62
1943–44	.32	.12	.33	.04	.81	.15	.10	.34	.04	.63	.20	.54	.74
1944–45	.34	.13	.34	.04	.85	.15	.10	.33	.06	.64	.20	.54	.74
1945–46	.46	.13	.36	.05	1.00	.18	.10	.34	.07	.69	.17	.55	.72
1946–47	.40	.12	.40	.05	.97	.16	.10	.39	.07	.72	.19	.54	.73
1947–48	.38	.13	.44	.06	1.01	.18	.09	.43	.08	.78	.18	.55	.73
1948–49	.38	.12	.46	.07	1.03	.20	.09	.42	.07	.78	.18	.59	.77
1949–50	.24	.13	.46	.08	.91	.17	.10	.46	.08	.81	.19	.57	.76
1950–51	.42	.12	.46	.09	1.09	.16	.10	.44	.09	.79	.22	.59	.81
1951–52	.35	.13	.51	.10	1.09	.15	.12	.49	.10	.86	.22	.63	.85
1952–53	.33	.15	.49	.08	1.05	.15	.11	.51	.08	.85	.23	.65	.88
1953–54	.49	.14	.54	.09	1.26	.18	.10	.48	.09	.85	.23	.66	.89
1954–55	.41	.14	.48	.09	1.12	.20	.09	.45	.09	.83	.27	.65	.92
1955–56	.40	.15	.48	.10	1.13	.27	.10	.45	.10	.92	.26	.64	.90
1956–57	.44	.15	.50	.10	1.19	.26	.11	.44	.10	.91	.31	.68	.99
1957–58	.37	.17	.56	.14	1.24	.33	.13	.44	.12	1.02	.36	.70	1.06
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	.45	.14	.40	.10	1.18	.25	.74	.99

1960–61	.55	.20	.54	.13	1.42	.51	.12	.44	.13	1.20	.29	.74	1.03
1961–62	.68	.23	.54	.13	1.58	.55	.13	.44	.13	1.25	...§	...	...
1962–63	.59	.24	.52	.14	1.49	.67	.13	.44	.14	1.38	...	...	....
1963–64	.56	.26	.53	.12	1.47	.42	.19	.54	.12	1.27	...	...	...
1964–65	.60	.24	.54	.13	1.51	.50	.23	.53	.13	1.39	...	...	....
1965–66	.60	.28	.55	.12	1.55	.46	.23	.57	.12	1.38	...	...	...
1966–67	.58	.29	.55	.14	1.56	.65	.23	.57	.14	1.59	...	...	...
1967–68	.81	.29	.64	.21	1.95	.46	.26	.60	.21	1.53	...	...	...
Five-Year Averages													
39–40 through 43–44	.26	.08	.27	.04	.65	.13	.07	.27	.04	.51	.19	.43	.62
44–45 through 48–49	.39	.13	.40	.05	.97	.17	.10	.38	.07	.72	.18	.55	.73
49–50 through 53–54	.37	.13	.49	.09	1.08	.16	.11	.48	.09	.84	.22	.62	.84
54–55 through 58–59	.39	.15	.50	.11	1.15	.30	.11	.45	.10	.96	.29	.68	.97
59–60 through 63–64	.61	.22	.53	.13	1.49	.52	.14	.46	.13	1.25	.31	.74	1.05

*From Sunkist Growers, Inc. (1967, 1969).

†The Florida year begins in September. Cultural costs for all areas refer to costs incurred in producing the crop marketed during a given season, without regard to the time of expenditure.

‡The costs per carton were obtained by dividing the total cultural cost per acre (about 0.405 hectare), *exclusive of interest on investment and depreciation on trees*, by the total yield per acre.

§Blanks mean "data not available."

An estimate of the average cost per box for picking and hauling Florida grapefruit is recorded in table 24. These values were obtained and calculated from reports submitted by 30 to 37 selected firms for the crop seasons 1953–54 through 1963–64. During this decade, the average cost per box for picking and hauling increased by approximately 50 per cent.

When grapefruit is sold for fresh consumption, an additional marketing cost is added for packaging and selling. The data in table 25 show the average cost per standard box equivalent for packing and selling the fresh fruit. Over the 10-year period, these charges increased about 38 cents per carton and 25 cents per box, which amounted to approximately 41 and 30 per cent, respectively. All figures quoted in tables 24 and 25 are constantly changing with time, usually increasing with labor and other production costs that are related to the general inflationary rise in the cost of living.

A relative comparison of the cost of production and marketing of California–Arizona and Florida grapefruit can be made from the data reported in table 26. The cultural costs include the same items, and are often referred to as the grower's production costs. It can be seen that the average cultural costs in the California–Arizona area are significantly higher than those in Florida. Packing-house charges in California, such as picking, hauling, packing, and selling, are distinctly higher than those in Florida, but the production and marketing costs for California–Arizona desert grapefruit appear to be closer to the Florida figures. An examination of the columns for total cost FOB packing house for both California and Florida will show definitely that California has higher production and marketing costs for fresh grapefruit.

The data reported in table 27 show that 2,088 cars of Florida fresh grapefruit were sold at terminal market auctions during the 1968–69 season. About 91 per cent of this volume had been grown in the Indian River district. These auction sales represented a relatively small percentage of the total volume of 13.9 million boxes sold for fresh use in that crop season.

It should be noted that the prices received for the auction sales of fresh Florida grapefruit were far above the average over-all prices received for sales for fresh use (table 28). Furthermore, the average prices for all sales for fresh use were far above those for similar fruit diverted to processing plants. In some years during the 20-year period, the average on-tree price of grapefruit for processing purposes reached extremely low values of 11 and 12 cents per box. The time-course curves in figure 10 graphically illustrate the differences in prices received for both fresh and processed Florida grapefruit between 1955 and 1965.

Additional data on the Florida crop show the seasonal average on-tree price, distributed according to utilization of sales as well as by area of production and horticultural type (table 29). During the 9-year period from 1960–61 through 1968–69, fruit from the Indian River area brought a higher

TABLE 27

CARS OF FLORIDA GRAPEFRUIT SOLD AND AVERAGE PRICES PAID AT TERMINAL MARKET AUCTIONS, REPORTED BY DISTRICTS, FOR THE CROP SEASONS 1954-55 THROUGH 1968–69.*

Crop seasons	All districts		Interior fruit		Indian River fruit		
	Average prices (dollars per box)	Volumes sold (cars)	Average prices (dollars per box)	Volumes sold (cars)	Average prices (dollars per box)	Volumes sold	
						Quantities (cars)	Percentages of all districts
1954–55	3.70	8,677	3.15	3,639	4.10	5,038	58.1
1955–56	4.01	7,361	3.44	2,666	4.33	4,695	63.8
1956–57	4.41	6,647	3.73	2,143	4.73	4,504	67.8
1957–58	4.91	5,714	3.87	1,454	5.25	4,260	74.6
1958–59	4.39	5,963	4.06	1,131	4.47	4,832	81.0
1959–60	4.41	4,941	4.02	961	4.50	3,980	80.6
1960–61	4.32	4,445	4.21	780	4.39	2,885	64.9
1961–62	4.49	3,773	4.06	561	4.57	3,212	85.1
1962–63	5.88	3,700	4.40	407	6.07	3,293	89.0
1963–64	6.09	3,050	5.59	494	6.15	2,556	83.8
1964–65	5.94	2,956	5.58	603	6.04	2,353	79.7
1965–66	5.82	2,531	4.86	340	6.00	2,191	87.0
1966–67	4.74	3,123	4.68	461	4.74	2,662	85.2
1967–68	6.48	2,237	5.43	224	6.61	2,013	90.0
1968–69	5.60	2,088	5.64	179	5.60	1,909	91.4

*From Florida Department of Agriculture (1969).

price per box, when sold for fresh use, than fruit from similar varieties obtained from the interior areas. Apparently, the area of production did not affect the price of fruit that was destined for the processor, but comparisons within varieties showed that the seedy type commanded the highest price per box for fruit diverted to processing. The pink seedless had the highest state average for all sales, which is the weighted average for both fresh use and processing. The price of fresh fruit from the 2 areas fluctuated significantly over the 9-year period. [*See pages 56 through 61 for tables 29 through 32.*]

The on-tree prices of grapefruit sold for processing in California are recorded in table 30. During the 29 crop seasons, 1939–40 through 1967–68, the highest price paid was 45 cents per packed equivalent carton, and in some years losses occurred to the growers. With such low financial returns on grapefruit going to the processor, it is obvious that the sale of grapefruit for

TABLE 28

SEASONAL AVERAGE ON-TREE PRICES OF FLORIDA GRAPEFRUIT PAID PER BOX, AS UTILIZED FOR FRESH USE AND PROCESSING, FOR THE CROP SEASONS 1948–49 THROUGH 1967–68.*

(Shown in dollars and cents.)

Crop seasons	For fresh use	For processing	Averages of all sales
1948–49	0.95	0.43	0.67
1949–50	1.99	1.63	1.79
1950–51	1.22	.70	.94
1951–52	.81	.12	.52
1952–53	1.08	.40	.76
1953–54	.86	.11	.49
1954–55	.95	.24	.63
1955–56	.92	.20	.57
1956–57	1.36	.44	.89
1957–58	1.37	.63	.98
1958–59	1.36	.75	1.04
1959–60	1.31	.76	1.05
1960–61	1.24	.66	.96
1961–62	1.06	.26	.67
1962–63	2.02	.55	1.24
1963–64	2.62	1.78	2.24
1964–65	2.03	.92	1.47
1965–66	1.87	.97	1.36
1966–67	1.21	.44	.75
1967–68	2.52	1.29	1.83

*From Florida Department of Agriculture (1969).

processing purposes will not pay the relatively high costs of production and marketing that exist under California conditions.

The citrus industry went through a period of rapid expansion before World War II. Since that time, production has continued to expand in some areas and level off in others, but revolutionary changes have taken place in

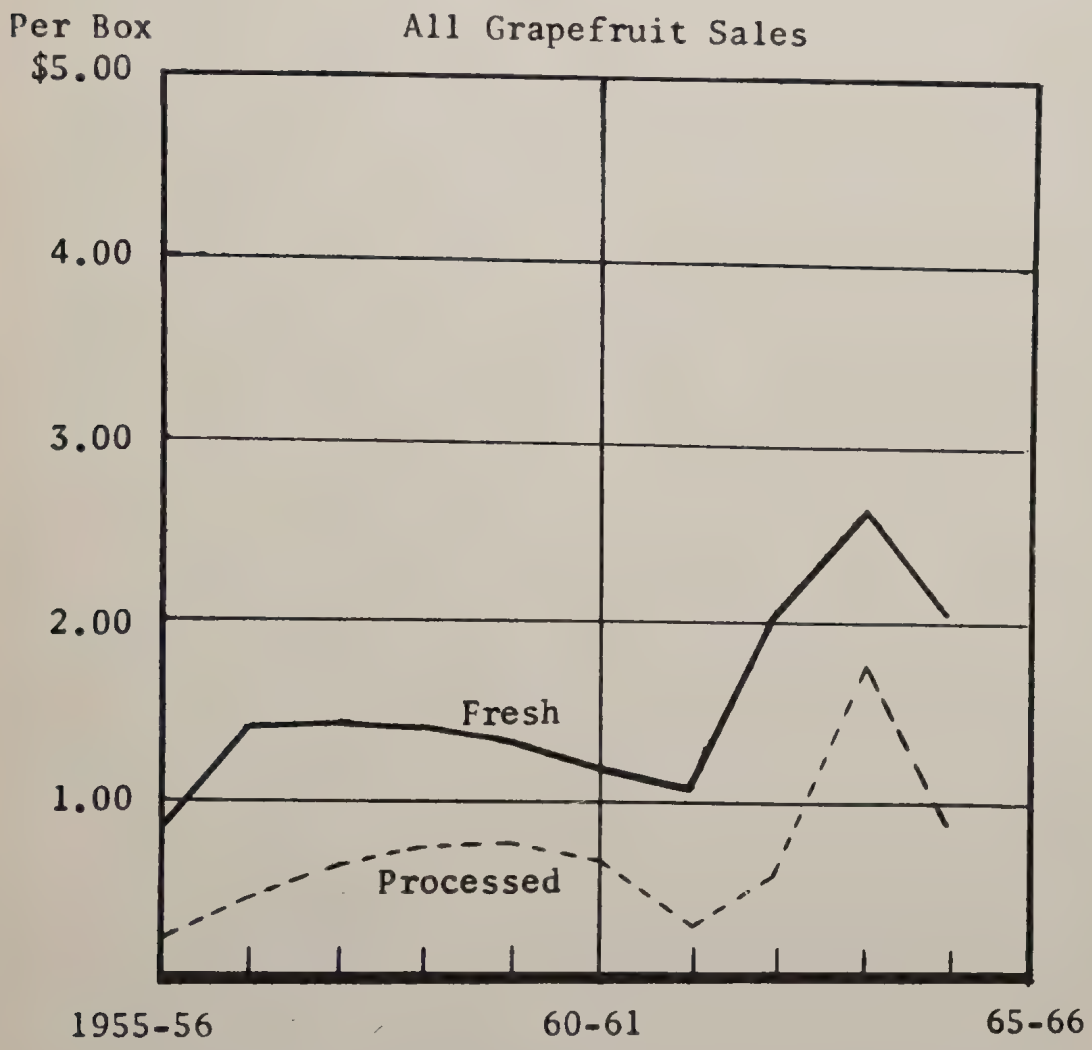


Fig. 10. Variations in seasonal average on-tree prices per box for both fresh and processed grapefruit, 1955–56 through 1964–65. (From Florida Department of Agriculture, 1965.)

the utilization and marketing of all citrus fruits. With worldwide increase in the production of grapefruit and other commercial varieties of citrus, a greater volume of the annual crop will be diverted to processed products and subsequently placed in storage for future sale. Great changes have also occurred in the marketing of fresh grapefruit. As just noted, only a small volume of Florida's fresh grapefruit sales has been made through the auction markets, although at a somewhat earlier period, nearly all citrus fruit for

fresh use was sold at auction in the terminal markets. With expanded transportation facilities, especially in the trucking field, and with the increased use of refrigeration in both truck and rail facilities, quicker sales and faster deliveries can be made directly to buyers without selling by auction.

Table 31 reveals some interesting relationships in connection with the production, utilization, and economic value of grapefruit crops grown in various United States areas over the 10-year period, 1959–69. Using data for the crop season 1968–69 (Section A), the seasonal average on-tree price per box for all sales may be estimated by dividing the total monetary return from all sales (49.5 million dollars) by the total number of boxes (53.8 million) sold for both fresh and processed uses. According to this calculation, the seasonal average figure amounted to 92 cents per box. The seasonal average amount received for fruit diverted to fresh use was $1.88 per box. This value may be derived by dividing the total fresh-fruit sales (41.7 million dollars) by the total number of boxes (22.2 million) sold through fresh-fruit channels. In 1968–69, the 31.6 million boxes of grapefruit that were processed had a value of over 7.8 million dollars, yielding a seasonal average price of 25 cents per box.

The value of the Florida grapefruit crop for 1968–69 (Section B) amounted to 31.6 million dollars, and this was equivalent to approximately 63.8 per cent of the total dollar value for all grapefruit-producing areas in the United States (Section A). From fresh-fruit sales, the Florida industry received 24.3 million dollars, which was equivalent to 58.3 per cent of the total fresh-fruit sales volume of 41.7 million dollars. In terms of utilization, Florida sold 13.9 million boxes in 1968–69 through the fresh-fruit market, which represented 62.6 per cent of the total United States volume of fresh grapefruit.

Over the 10-year period, the seasonal average price of grapefruit for processing amounted to 70 cents per box for all producing areas, but the average price paid for fruit destined for processing varied considerably between areas. In Florida, the average was 79 cents per box, while in California it was 21 cents, in Texas 63 cents, and in Arizona 18 cents. Such low prices received for grapefruit that is diverted to the processor do not provide a profitable return to the majority of growers. In the 1968–69 season, Florida processed 25.8 million boxes (about 65 per cent of the total utilizable production) at a seasonal average price of 29 cents per box. This was equivalent to 2.9 times the lowest average price received in Arizona for similar fruit (10 cents per box). However, the price paid for processing grapefruit in Florida during 1968–69 was much lower (29 cents per box) than the 10-year average (79 cents per box) for that state.

In 1968–69, the total value of grapefruit sales for all producing areas of the United States was 49.5 million dollars, including fruit for both fresh and processing uses. The total value of the 1968–69 crop for California, Texas,

and Arizona amounted to 17.9 million dollars, and this amount was distributed among the 3 producing areas as follows: California, 7.0; Texas, 7.4; and Arizona, 3.5 million dollars. The returns from California grapefruit were derived solely from the sale of fresh fruit, of which only 46.9 per cent of the total production of 4.9 million boxes was diverted through fresh-fruit channels. Conversely, 2.6 million boxes of California grapefruit (53.1 per cent of the total production) were processed at a cost to the growers of 431,000 dollars (Section C). More than 90 per cent of the total sales value of Texas and Arizona grapefruit was derived from fresh-fruit sales. Texas marketed 4.5 million boxes of grapefruit for fresh use, which was equivalent to 68.2 per cent of the state's total production. Arizona diverted 1.4 million boxes to the fresh-fruit market, which accounted for 56 per cent of the state's total production of 2.5 million boxes.

United States per Capita Consumption. The per capita consumption of fresh grapefruit in the United States is shown in table 32. For comparison, figures for oranges, tangerines, lemons, and other fruits are shown. From 1944–45, there was a downward trend in the consumption of all fresh citrus fruits, and from this period to 1962–63 the per capita consumption declined from 66.6 to 22.1 pounds, a decrease of 66.8 per cent. Specifically, grapefruit declined from 13.5 to 6.4 pounds per capita, a drop of approximately 52.6 per cent. During this period, the utilization of fresh bananas and apples remained practically constant. In the crop season 1962–63, the per capita consumption of grapefruit was 6.4 pounds, and this amount was equivalent to 28.9 per cent of all fresh citrus fruit consumed in that period. Another relationship for the same period was that all fresh citrus consumed amounted to 29 per cent of the total fresh fruit used.

In the last 3 columns of table 32, the comparative production of oranges, lemons, and grapefruit is recorded in terms of the number of fruits produced per capita. For all these citrus varieties, the number of fruits per person has shown a tendency to decrease, with occasional fluctuations to the contrary. A considerable portion of this decrease is primarily owing to the population growth in the United States. For grapefruit, particularly, the number produced per capita dropped from 24 to 11.3 between 1944–45 and 1963–64. Another interesting comparison shows that there were nearly 9 times more oranges and 2 times more lemons produced per capita than grapefruit.

The reduction in consumption of fresh grapefruit, as well as of oranges and lemons, has taken place concurrently with a large increase in total world production and in the use of processed citrus products. The fresh-fruit consumption per capita shows a downward trend for nearly all fruits in table 32. The total fresh fruit per person dropped from 139.9 pounds to 74.4 pounds during the period from 1944–45 through 1962–63, a decrease of about 46.8 per cent for that particular interval.

TABLE 29

SEASONAL AVERAGE ON-TREE PRICES OF 4 TYPES OF FLORIDA GRAPEFRUIT PAID PER BOX, REPORTED BY UTILIZATION OF FRUIT AND AREAS OF PRODUCTION, FOR THE CROP SEASONS 1960–61 THROUGH 1968–69.*

(Shown in dollars and cents.)

Crop seasons and areas	White seedless			Pink seedless			All seedless			Seedy		
	Fresh use	Processing	All sales	Fresh use	Processing	All sales	Fresh use	Processing	All sales	Fresh use	Processing	All sales
1960–61												
Interior	0.92	0.55	0.76	0.82	0.18	0.70	0.88	0.49	0.74	1.04	0.78	0.83
Indian River	2.07	.55	1.75	1.65	.18	1.33	1.86	.36	1.54	1.70	.78	1.00
State averages	1.34	.55	1.05	1.19	.18	.99	1.27	.46	1.03	1.08	.78	.84
1961–62												
Interior	.83	.14	.51	.95	−.13	.68	.88	.09	.57	.82	.43	.50
Indian River	1.47	.14	1.16	1.36	−.13	1.10	1.41	.02	1.13	1.35	.43	.65
State averages	1.05	.14	.68	1.13	−.13	.86	1.09	.07	.75	.87	.43	.51
1962–63												
Interior	1.23	.54	.75	1.28	.24	1.04	1.25	.51	.84	1.08	.62	.71
Indian River	2.75	.54	2.25	2.79	.24	2.36	2.77	.41	2.30	2.45	.62	1.03
State averages	2.11	.54	1.29	2.19	.24	1.81	2.15	.49	1.49	1.22	.62	.74
1963–64												
Interior	2.29	1.72	2.02	2.50	1.47	2.23	2.37	1.67	2.08	1.91	1.91	1.91
Indian River	2.94	1.72	2.69	3.00	1.47	2.77	2.97	1.61	2.73	2.08	1.91	1.95
State averages	2.56	1.72	2.24	2.80	1.47	2.54	2.67	1.66	2.36	1.94	1.91	1.91
1964–65												
Interior	1.57	.85	1.20	1.60	.60	1.31	1.58	.79	1.24	1.43	1.05	1.11
Indian River	2.59	.85	2.20	2.77	.60	2.35	2.68	.73	2.28	1.87	1.05	1.32
State averages	1.98	.85	1.50	2.21	.60	1.82	2.08	.78	1.63	1.51	1.05	1.13
1965–66												
Interior	1.52	.96	1.17	1.58	.66	1.29	1.55	.90	1.21	1.29	1.06	1.08
Indian River	2.27	.96	1.94	2.34	.66	2.01	2.31	.82	1.98	1.51	1.06	1.20
State averages	1.85	.96	1.39	1.98	.66	1.64	1.91	.89	1.49	1.33	1.06	1.09
1966–67												
Interior	.96	.40	.58	1.08	.11	.75	1.02	.35	.63	.89	.57	.60
Indian River	1.45	.40	1.16	1.55	.11	1.17	1.50	.25	1.17	.89	.57	.74
State averages	1.16	.40	.72	1.30	.11	.94	1.24	.33	.81	.89	.57	.60
1967–68												
Interior	2.52	1.22	1.75	2.62	.66	2.10	2.57	1.12	1.86	1.95	1.43	1.47
Indian River	3.44	1.22	2.58	3.59	.66	2.81	3.52	.99	2.69	1.96	1.43	1.60
State averages	2.94	1.22	2.05	3.14	.66	2.48	3.04	1.08	2.22	1.95	1.43	1.48
1968–69												
Interior	1.25	.26	.54	1.35	−.14	.79	1.30	.18	.62	1.54	.46	.52
Indian River	2.17	.26	1.40	2.22	−.14	1.31	2.19	.07	1.36	1.52	.46	.72
State averages	1.72	.26	.82	1.76	−.14	1.04	1.74	.15	.90	1.54	.46	.53

*From Florida Department of Agriculture (1969).

TABLE 30

ON-TREE PRICES PAID FOR CALIFORNIA–ARIZONA AND OTHER UNITED STATES GRAPEFRUIT IN THE CROP SEASONS 1939–40 THROUGH 1967–68.*
(Shown in dollars and cents per packed equivalent carton.)

Crop seasons	California summer			California–Arizona desert			Other United States grapefruit		
	Fresh	Processed	All uses	Fresh	Processed	All uses	Fresh	Processed	All uses
1939–40	0.35	0.03	0.34	0.14	0.06	0.12	0.22	0.10	0.17
1940–41	.53	.01	.49	.14	.02	.12	.21	.12	.17
1941–42	.80	.01	.71	.32	.09	.25	.36	.23	.30
1942–43	.99	.11	.94	.65	.31	.50	.59	.42	.49
1943–44	1.22	.01	1.06	.76	.42	.61	.73	.63	.67
1944–45	1.21	.09	1.04	.91	.32	.71	.81	.70	.76
1945–46	.88	.02	.64	.62	.25	.47	.69	.52	.59
1946–47	.85	−.12	.62	.29	−.06	.15	.49	.19	.34
1947–48	.64	−.19	.42	.39	−.05	.24	.29	.07	.17
1948–49	.95	−.12	.77	.59	−.02	.28	.48	.18	.33
1949–50	.86	−.02	.58	.80	.08	.30	.97	.68	.82
1950–51	.74	−.09	.48	.43	.11	.30	.61	.31	.45
1951–52	1.02	−.06	.61	.59	.02	.39	.46	.05	.29
1952–53	.95	−.08	.58	.62	.09	.42	.58	.19	.40
1953–54	1.01	−.06	.68	.69	.07	.44	.49	.06	.29
1954–55	1.29	−.08	.87	.61	.06	.41	.54	.12	.36
1955–56	1.38	.03	.92	.72	.08	.50	.53	.10	.34
1956–57	1.17	.00	.79	.89	.07	.61	.71	.21	.47
1957–58	1.56	.17	1.27	.86	.19	.76	.76	.30	.55
1958–59	1.09	.07	.77	.92	.08	.74	.73	.36	.55
1959–60	1.04	.07	.80	.73	.04	.48	.67	.34	.53
1960–61	.94	.08	.73	.67	.03	.43	.62	.29	.48
1961–62	.97	−.02	.67	.59	.01	.35	.56	.12	.36
1962–63	1.99	.11	1.41	1.06	.01	.52	1.07	.25	.64
1963–64	1.66	.45	1.27	1.24	.32	.92	1.33	.79	1.10
1964–65	1.17	−.24	.76	.90	.08	.56	1.02	.41	.74
1965–66	1.61	.16	1.10	1.03	.21	.69	.98	.45	.71
1966–67	1.44	.09	1.03	1.00	.12	.59	.68	.21	.42
1967–68	1.80	.10	1.15	1.18	.21	.82	1.24	.58	.91
Five-year averages									
39–40 through 43–44	.78	.03	.71	.40	.18	.32	.42	.30	.36
44–45 through 48–49	.91	−.06	.70	.56	.09	.37	.55	.33	.44
49–50 through 53–54	.92	−.06	.59	.63	.07	.37	.62	.26	.45
54–55 through 58–59	1.30	.04	.92	.80	.10	.60	.65	.22	.45
59–60 through 63–64	1.32	.14	.98	.86	.08	.54	.85	.36	.62

*From Sunkist Growers, Inc. (1967, 1969).

TABLE 31

GRAPEFRUIT: PRODUCTION, UTILIZATION, SEASONAL AVERAGE ON-TREE PRICES PAID PER BOX, AND VALUES OF SALES, OVER-ALL AND BY STATES, FOR THE CROP SEASONS 1959–60 THROUGH 1968–69.*

Crop seasons	Commercial production (1,000 boxes)‡	For farm home use (1,000 boxes)	Utilization of sales			Seasonal average prices†			Values of sales		
			Fresh use (1,000 boxes)	Processing (1,000 boxes)	Totals (1,000 boxes)	Fresh use (dollars per box)	Processing (dollars per box)	All sales (dollars per box)	Fresh use (1,000 dollars)	Processing (1,000 dollars)	Totals (1,000 dollars)
SECTION A: UNITED STATES (FLORIDA, CALIFORNIA, TEXAS, AND ARIZONA)											
1959–60	41,591	277	23,823	17,491	41,314	1.34	0.67	1.06	31,919	11,790	43,709
1960–61	43,370	277	24,345	18,748	43,093	1.24	.58	.95	30,198	10,842	41,040
1961–62	42,490	272	23,170	19,048	42,218	1.11	.23	.71	25,687	4,455	30,142
1962–63	34,738	192	16,449	18,097	34,546	2.14	.49	1.28	35,199	8,932	44,131
1963–64	34,207	267	19,781	14,159	33,940	2.65	1.58	2.20	52,370	22,406	74,776
1964–65	41,027	267	21,849	18,911	40,760	2.04	.82	1.47	44,499	15,487	59,986
1965–66	46,695	292	22,709	23,694	46,403	1.96	.89	1.41	44,539	21,143	65,682
1966–67	55,676	297	24,941	30,438	55,379	1.35	.41	.84	33,642	12,568	46,210
1967–68	44,058	299	21,932	21,827	43,759	2.78	1.11	1.95	60,951	24,255	85,206
1968–69	54,070	288	22,179	31,603	53,782	1.88	.25	.92	41,703	7,823	49,526
SECTION B: FLORIDA											
1959–60	30,500	160	16,032	14,308	30,340	1.31	.76	1.05	21,009	10,900	31,909
1960–61	31,600	160	15,726	15,714	31,440	1.24	.66	.96	19,562	10,394	29,956
1961–62	34,800	180	17,811	16,809	34,620	1.06	.26	.67	18,892	4,428	23,320
1962–63	30,000	125	13,913	15,962	29,875	2.02	.55	1.24	28,100	8,825	36,925
1963–64	26,300	180	14,539	11,581	26,120	2.62	1.78	2.24	38,089	20,616	58,705
1964–65	31,900	180	15,666	16,054	31,720	2.03	.92	1.47	31,736	14,812	46,548
1965–66	34,900	180	14,897	19,823	34,720	1.87	.97	1.36	27,848	19,310	47,158
1966–67	43,600	180	17,101	26,319	43,420	1.21	.44	.75	20,607	11,584	32,191
1967–68	32,900	180	14,522	18,198	32,720	2.98	1.24	2.01	43,269	22,559	65,828
1968–69	39,900	175	13,892	25,833	39,725	1.75	.29	.80	24,274	7,372	31,646

SECTION C: CALIFORNIA

1959–60	2,671	22	1,803	846	2,649	2.01	.00	1.37	3,616	3	3,619
1960–61	2,630	22	1,720	888	2,608	1.76	−.06	1.15	3,036	−58	2,978
1961–62	2,820	22	1,790	1,008	2,798	1.83	−.02	1.17	3,282	−17	3,265
1962–63	2,498	22	1,601	875	2,476	3.44	.17	2.29	5,511	145	5,656
1963–64	4,197	22	2,683	1,492	4,175	3.18	.98	2.40	8,537	1,460	9,997
1964–65	4,227	22	2,579	1,626	4,205	2.52	.28	1.65	6,499	452	6,951
1965–66	4,945	22	2,899	2,024	4,923	2.96	.43	1.92	8,594	874	9,468
1966–67	4,996	22	2,945	2,029	4,974	2.54	.23	1.60	7,471	471	7,942
1967–68	4,618	24	2,820	1,774	4,594	3.20	.26	2.07	9,025	465	9,490
1968–69	4,960	16	2,344	2,600	4,944	3.16	−.17	1.41	7,414	−431	6,983

SECTION D: TEXAS

1959–60	5,200	75	3,881	1,244	5,125	1.19	.59	1.04	4,618	734	5,352
1960–61	6,800	75	5,444	1,281	6,725	1.07	.24	.91	5,825	307	6,132
1961–62	2,700	50	2,294	356	2,650	1.07	.05	.93	2,455	18	2,473
1962–63	70	25	45	.....	45	4.04	....	4.04	182	...	182
1963–64	500	45	430	25	455	3.21	1.78	3.13	1,380	44	1,424
1964–65	2,000	45	1,816	139	1,955	2.12	1.13	2.05	3,850	157	4,007
1965–66	3,800	70	3,010	720	3,730	1.71	.80	1.52	5,147	576	5,723
1966–67	5,400	80	3,960	1,360	5,320	.98	.27	.80	3,881	367	4,248
1967–68	2,800	80	2,205	515	2,720	1.98	1.09	1.82	4,364	561	4,925
1968–69	6,700	80	4,510	2,110	6,620	1.49	.30	1.11	6,720	633	7,353

SECTION E: ARIZONA

1959–60	3,220	20	2,107	1,093	3,200	1.27	.14	.88	2,676	153	2,829
1960–61	2,340	20	1,455	865	2,320	1.22	.23	.85	1,775	199	1,974
1961–62	2,170	20	1,275	875	2,150	.83	.03	.50	1,058	26	1,084
1962–63	2,170	20	890	1,260	2,150	1.58	−.03	.64	1,406	−38	1,368
1963–64	3,210	20	2,129	1,061	3,190	2.05	.27	1.46	4,364	286	4,650
1964–65	2,900	20	1,788	1,092	2,880	1.35	.06	.86	2,414	66	2,480
1965–66	3,050	20	1,903	1,127	3,030	1.55	.34	1.10	2,950	383	3,333
1966–67	1,680	15	935	730	1,665	1.80	.20	1.10	1.683	146	1,829
1967–68	3,740	15	2,385	1,340	3,725	1.80	.50	1.33	4,293	670	4,963
1968–69	2,510	17	1,433	1,060	2,493	2.30	.10	1.36	3,295	249	3,544

*From Florida Department of Agriculture (1969).
†Derived from the sums of sales and values of types or areas.
‡Standard boxes of 80 pounds (about 36 kilograms).

TABLE 32

UNITED STATES PER CAPITA CONSUMPTION OF FRESH CITRUS AND DECIDUOUS FRUITS, AND PER CAPITA PRODUCTION OF CITRUS FRUITS, 1939–40 THROUGH 1966–67.*

Crop seasons or years†	Per capita consumption (pounds)‡													Per capita production of citrus fruits (number of fruits)		
	Fresh citrus fruits				Fresh deciduous fruits								Totals, all fresh fruits			
	Oranges; tangerines	Lemons	Grapefruit	All citrus§	Apples‖	Bananas	Grapes	Peaches	Pears	Strawberries	Ten other fruits¶	All deciduous		Oranges; tangerines	Lemons	Grapefruit
1939–40	41.0	4.5	11.1	56.7	29.7	17.3	6.3	13.1	7.1	3.3	5.6	82.4	139.1	105.5	30.1	17.2
1940–41	40.7	4.7	12.2	57.7	31.7	16.6	6.2	18.6	6.4	3.1	5.7	88.3	146.0	118.0	42.7	20.7
1941–42	41.2	4.3	12.1	57.7	28.1	8.0	6.2	14.6	6.7	3.4	5.3	72.3	130.0	116.0	28.7	19.2
1942–43	42.6	5.0	12.5	60.3	24.9	6.9	5.6	8.4	5.4	1.8	5.1	58.1	118.4	120.4	36.1	23.8
1943–44	50.1	4.9	13.0	68.2	25.5	9.0	4.9	17.9	7.1	1.2	6.3	61.9	140.1	142.0	26.6	26.1
1944–45	47.8	5.1	13.5	66.6	22.9	12.1	5.6	18.2	7.3	1.3	5.9	73.3	139.9	148.8	29.8	24.0
1945–46	40.3	4.7	14.0	59.1	23.0	14.7	5.7	16.6	6.8	1.6	6.4	74.8	133.9	135.8	34.0	28.9
1946–47	43.4	4.8	13.9	62.2	25.4	21.5	6.6	14.8	5.9	1.9	5.4	81.5	143.7	151.4	31.7	26.6
1947–48	37.5	4.5	12.3	54.4	26.3	22.4	5.8	11.3	4.4	1.8	5.2	77.2	131.6	143.7	29.1	27.1
1948–49	32.8	4.1	10.9	47.9	24.7	20.8	5.2	11.6	5.5	1.6	5.8	75.2	123.1	128.0	22.1	19.6
1949–50	28.9	4.0	8.2	41.3	22.7	20.9	5.4	7.8	4.1	1.6	4.8	67.3	108.6	131.2	24.8	15.4
1950–51	30.7	4.0	10.3	45.1	25.7	20.5	5.9	9.4	4.0	1.8	5.0	72.3	117.4	144.5	28.6	19.4
1951–52	29.9	3.9	10.5	44.4	21.6	20.6	6.0	10.7	4.4	1.6	4.5	69.4	113.8	143.0	26.9	16.5
1952–53	29.8	3.7	9.7	43.4	20.9	19.5	4.8	10.3	3.9	1.4	4.7	65.5	108.9	143.5	26.0	15.4
1953–54	26.5	3.6	11.0	41.2	20.0	18.9	5.1	10.0	3.7	1.2	4.3	63.2	104.4	147.3	32.6	19.0
1954–55	26.9	3.4	10.7	41.2	19.6	17.8	5.0	6.1	3.4	1.2	4.6	57.7	98.9	149.9	27.8	16.3
1955–56	24.5	3.2	10.5	38.5	18.9	18.0	4.7	9.0	3.7	1.5	4.1	59.9	98.4	148.7	25.8	17.2
1956–57	23.4	3.3	9.5	36.5	19.3	18.0	3.9	8.6	3.7	1.7	4.5	59.7	96.2	145.6	31.1	16.7
1957–58	18.5	3.0	8.7	30.5	22.6	17.2	4.1	10.5	3.5	1.5	3.8	63.2	93.7	116.5	31.8	14.6
1958–59	21.2	2.9	9.1	33.4	23.0	18.2	3.9	9.7	3.2	1.3	4.4	63.7	97.1	137.4	31.2	15.7
1959–60	20.5	2.9	10.0	33.7	18.2	20.5	3.9	9.5	2.6	1.3	3.6	59.6	93.3	130.6	31.0	14.7
1960–61	17.9	2.8	9.8	30.8	16.4	19.9	3.5	9.7	2.6	1.6	4.0	57.7	88.5	120.4	24.6	15.0
1961–62	17.2	2.8	9.0	29.5	17.4	16.3	4.0	8.1	2.6	1.6	3.8	53.8	83.3	138.5	26.7	14.6
1962–63	12.8	2.5	6.4	22.1	16.7	16.6	4.0	7.6	2.0	1.6	3.8	52.3	74.4	102.6	22.5	11.7
1963–64	15.6	2.6	7.5	26.1	17.8	16.8	3.6	6.0	2.4	1.6	4.3	52.5	78.6	91.2	32.4	11.3

1964–65	17.9	2.4	8.2	29.0	16.3	17.9	3.8	6.9	1.8	1.3	3.9	51.9	80.9	117.0	24.5	13.3
1965–66	17.8	2.3	8.4	29.0	15.9	18.3	3.8	6.3	2.4	1.3	4.2	52.2	81.2	147.5	23.7	19.3
1966–67	19.6	2.4	9.0	31.7	16.2	18.3	2.8	4.8	1.8	1.5	3.6	49.0	80.7	179.9	25.6	22.2
Five-year averages																
39–40 through 43–44	43.1	4.7	12.2	60.1	28.0	11.6	5.8	14.5	6.5	2.6	5.6	74.6	134.7	120.4	32.8	21.4
44–45 through 48–49	40.4	4.6	12.9	58.0	24.5	18.3	5.8	14.5	6.0	1.6	5.7	76.4	134.4	141.5	29.3	25.2
49–50 through 53–54	29.2	3.8	9.9	43.1	22.2	20.1	5.4	9.6	4.0	1.5	4.7	67.5	110.6	141.9	27.8	17.1
54–55 through 58–59	22.9	3.2	9.7	36.0	20.7	17.8	4.3	8.8	3.5	1.4	4.3	60.8	96.9	139.6	29.5	16.1
59–60 through 63–64	16.7	2.7	8.3	28.1	19.3	17.9	3.7	8.3	2.4	1.5	4.0	57.2	85.3	116.7	27.4	13.5

*From Sunkist Growers, Inc. (1967, 1969).
†Deciduous fruits are given on a calendar-year basis.
‡One pound equals 0.454 kilogram.
§Beginning 1947–48, "all citrus" includes limes. Beginning 1955–56, it includes tangelos.
‖Excluding noncommercial apples.
¶Apricots, avocados, cherries, cranberries, figs, nectarines, papayas, pineapples, plums, and prunes.

Chapter 2

Composition of the Edible Portions

GRAPEFRUIT RIPENS SLOWLY over a long period, and will hang on the tree for several months after it has reached edible quality. Composition is therefore important, not only for indicating nutritive values, but also for determining the proper time to harvest. A number of studies dealing with seasonal changes during the ripening period are reviewed in this chapter. Where not otherwise stated, the data are for mature grapefruit. Maturity standards in various grapefruit-growing areas are discussed in chapter 4.

The composition of the fruit varies, not only with the degree of maturity, but also with climate, soil, variety, cultural practices, and other factors. Climate exerts so strong an influence that the locality where the trees were grown must always be kept in mind when evaluating any data on composition. The period required for the fruit to mature and the time of year when it ripens differ widely in different climates—even in California, within a single state. Some differences caused by climate, variety, and methods of extraction are revealed by the data reviewed in this chapter. The effects of various factors on quality, maturity, and yield are discussed at some length in chapter 4, and changes that take place during storage are noted in chapter 5.

General Data on Composition

THE FRESH WEIGHT of mature grapefruit consists of 35 to 50 per cent juice, with the remainder made up of peel, rag, and seeds. Table 33 gives the seasonal changes in percentage of juice in 3 grapefruit varieties grown in Texas, as reported by Krezdorn and Cain (1952). The conditions of their experiment are described on pages 93–94 and in table 46.

The fresh juice consists of 88 to 93 per cent water, 8 to 13 per cent soluble solids, and small amounts of insoluble solids. Sugars (sucrose and reducing) and acids (chiefly citric) constitute 85 to 90 per cent of the soluble solids; the other solids, soluble and insoluble, are mainly vitamins, mineral salts, amino acids, proteins, fats, pectin, and glycosides.

Hawkins and Magness (1920) were among the early workers in grapefruit composition. They analyzed "Florida common" fresh grapefruit, and obtained the following ranges of values, stated as percentages of the wet weight

of pulp: dry matter, 7.94 to 8.96; reducing sugars, 2.16 to 2.95; sucrose, 1.66 to 2.82; total sugars, 4.32 to 5.43; acids, 0.93 to 1.28. For further results, see Hawkins (1921).

Specific Gravity of Fresh Whole Fruit. The specific gravity of the whole fruit gives useful information regarding its quality, because quality depends mainly upon the juice content and the amount of soluble solids, both of which affect specific gravity.

Haas and Klotz (1935) found that the specific gravity was the same in the stem and stylar ends of immature grapefruit, but greater in the stylar end of the mature fruit.

Stahl and Camp (1936) found the specific gravity of Florida-grown Marsh grapefruit picked on April 6 to be 0.8672. (For their data on changes in specific gravity during storage, see tables 168 and 169, chapter 5.)

Turrell and Slack (1948) determined the specific gravity of freshly picked, mature Washington Navel and Valencia oranges, Eureka and Lisbon lemons, and Marsh grapefruit grown in different climatic areas—coastal, desert, and intermediate—in California. They found the average specific gravity of the Marsh variety to be 0.824 in an interior valley and 0.789 in a desert area, but the difference was statistically nonsignificant. The tests were made at such times that the fruits from the 2 areas were at the same stage of maturity. The investigators were careful to choose fruits of the same size from each area because they had found that there was a significant negative correlation between size of fruit and specific gravity. This fact is demonstrated graphically in figure 11, which shows the correlation between specific gravity and fruit weight.

Specific gravity is defined as the ratio of the weight of a definite volume of a substance to the weight of an equal volume of water. The specific gravity of a citrus fruit is usually stated as the ratio of the mass to the volume, which gives a result that is satisfactory for most practical purposes, since the volume

TABLE 33

SEASONAL CHANGES IN PERCENTAGE OF JUICE IN 3 GRAPEFRUIT VARIETIES GROWN IN TEXAS.*

Dates, 1950	Percentage of juice by weight		
	Marsh	Thompson	Webb (Redblush)
August 21	33.8	33.2	33.8
September 18	42.7	40.1	41.7
October 16	47.2	44.6	45.8
November 13	47.7	49.4	50.0
December 11	49.1	49.1	48.8

*From Krezdorn and Cain (1952). (Refer also to table 46.)

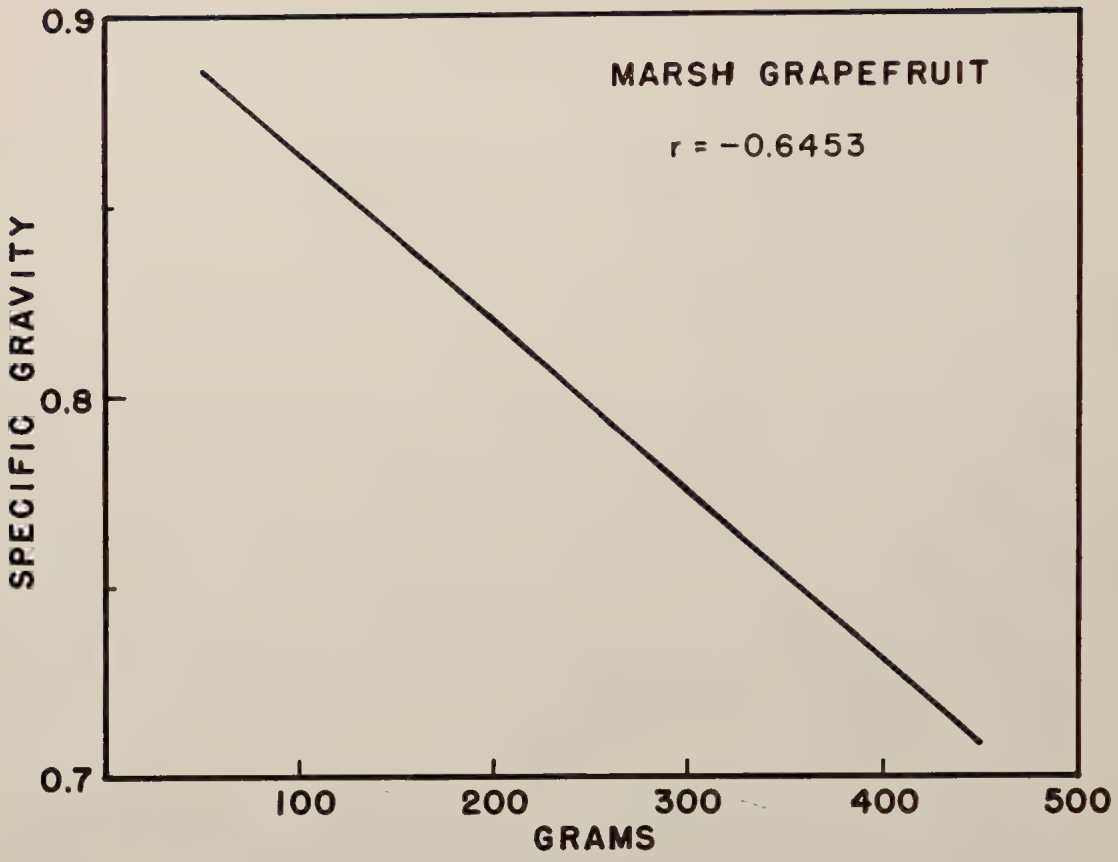


Fig. 11. The relationship of specific gravity to fruit weight. (From Turrell and Slack, 1948.)

of the fruit in milliliters is approximately equal to the weight of the volume of water displaced. Specific gravity has been found, in many instances, to correlate to a high degree with the chemical components and physical properties of various fruits and vegetables, such as pears, prunes, potatoes, and commercial varieties of citrus fruits. Water-flume separators, using the principle of specific gravity, have been used in the citrus industry for decades to separate mechanically the frozen from the nonfrozen fruit. Although frost-

TABLE 34

Correlation Coefficients Between Specific Gravity and Various Juice Characteristics of Valencia Oranges from the East Coast (Indian River) Area of Florida, May, 1963.*

Factors	Correlation coefficients
Specific gravity and percentage of juice.....	0.482**
Specific gravity and degrees Brix.....	.506**
Specific gravity and pounds of solids per box....	.801**
Specific gravity and total acidity.....	.283**

*From Ting and Blair (1965).
**Significant at the 1 per cent level.

damaged citrus fruits usually have a lower specific gravity than sound ones, Chace (1919) noted that the relationship between frost damage and specific gravity was not a perfect correlation. Grierson and Hayward (1959) confirmed and extended Chace's results.

Based on experimental data derived from Valencia orange samples, the correlation of specific gravity of the whole fruit with the juice characteristics is reported in table 34. Both the percentage of juice and the soluble solids (degrees Brix) were significantly correlated with specific gravity at the 1 per cent level. The correlation coefficient between specific gravity and pounds of solids per box was the highest value recorded (0.801). The titratable acidity of the juice was significantly correlated with the specific gravity of the whole fruit at a relatively low correlation coefficient (0.283).

The regression of pounds of solids per box on the specific gravity of multiple-fruit samples indicates the possibility of predicting the pounds of solids by measuring the average specific gravity of the fruit (figure 12). The regression line had a slope of 32.27, and the correlation coefficient was 0.902. Ting and Blair (1965) reported that the confidence limit for a single sample was more than 1 pound (0.454 kilogram) of solids per box from the expected

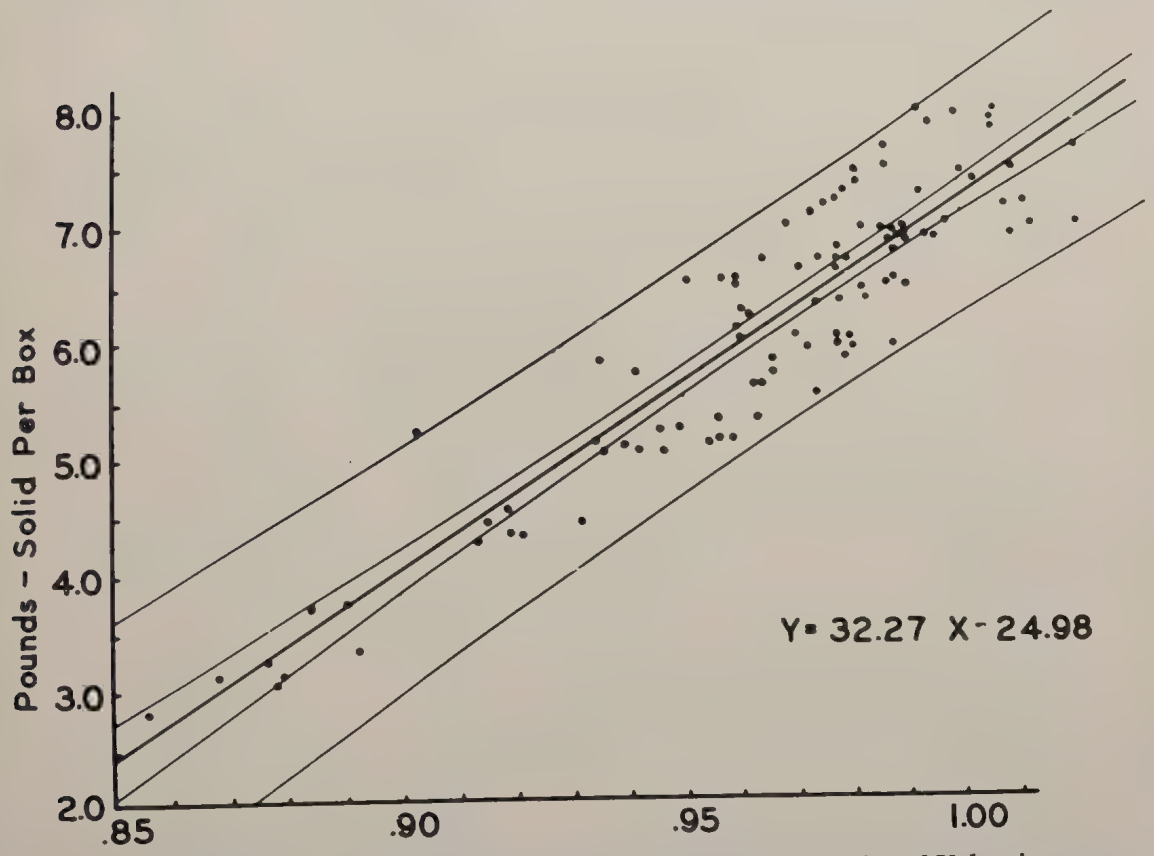


Fig. 12. Regression of pounds of solids per box on the specific gravity of Valencia oranges, February–May, 1965. The 95 per cent confidence lines were drawn on either side of the regression line. The 2 lines farthest from the regression line are the confidence intervals for individual samples. (From Ting and Blair, 1965.)

value calculated from the regression line, while the confidence limit (19 to 1) for the population mean was about 0.2 pound. The scatter diagram shown in figure 12 represents fruit of different sizes. Fruit size 125 had specific gravity values below 0.91; sizes 163 and 200 had values between 0.91 and 0.96; and the smallest sizes, 242 and 324, had values above 0.96.

Varietal Differences. Roberts and Gaddum (1937) included in their analyses not only the organic but also the inorganic constituents of the juice from 2 varieties of grapefruit, Marsh and a seedy variety. Each test sample consisted of half a box. All juice was extracted by hand, and an effort was made to avoid getting peel oil into it. The results are shown in table 35.

In several tests, Traub *et al.* (1929) determined the composition of the juice of 4 varieties of grapefruit grown in Texas. The fruits used in these tests were usually stored for 36 to 48 hours at 40 to 45° F. (4.4 to 7.2° C.) before the juice was extracted and tested. Some of the values obtained are shown in table 36. The greatest difference between varieties was in the number of seeds per fruit. From late August to mid-December, the 4 varieties showed: (1) only a slight decrease in soluble solids, a little more in acid; (2) single or total changes not enough to alter palatability; (3) ratios of soluble solids to total acids ranging from 7 to 9.

Fudge (1938) reported that whole fruits of a seedy variety contained slightly higher percentages of dry matter, total nitrogen, total ash, potassium, phosphorus, calcium, magnesium, iron, and manganese, but less aluminum than comparable fruits of the Marsh variety. From these results he concluded that the seedy varieties had a higher food value than the seedless ones.

TABLE 35

ANALYSIS OF GRAPEFRUIT JUICE.*

Constituents and related data	Marsh Seedless grapefruit	Seedy grapefruit
Citric acid, per cent	0.89	1.35
Specific gravity, 20°/20° C	1.028	1.039
Degrees Brix	6.95	9.74
Maturity ratio	7.81	7.22
pH	3.0	3.0
Water, per cent	93.05	90.26
Organic matter, per cent	6.73	9.33
Nitrogen, organic, per cent	0.043	0.052
Protein (N × 6.25), per cent	0.27	0.33
Reducing sugar, per cent	3.44	3.96
Sucrose, per cent	1.34	2.24
Total sugar, per cent	4.78	6.20
Pectic acid, per cent	0.004	0.004
Ash, per cent	0.218	0.414

*Adapted from Roberts and Gaddum (1937), their table 2.

TABLE 36

Composition of 4 Grapefruit Varieties Grown in Texas.*

Variety	Average weight (grams)			Peel thickness (mm)	Seeds (number)	Constituents of juice (percentages of fresh weight)				pH
	Fruits	Juice	Rag			Soluble solids	Total sugars	Total acids	Protein (N × 6.25)	
Marsh	466.2	255.3	96.1	6.0	3.9	10.3	6.68	1.14	0.59	3.18
Thompson	449.3	264.8	83.6	5.4	6.5	9.43	....	1.09	....	3.27
Foster	521.1	296.7	96.7	5.1	54.7	10.48	....	1.18	....	3.29
Duncan	468.5	241.5	105.8	6.2	63.0	9.47	....	1.09	....	3.30

*Adapted from Traub *et al.* (1929).

General Values for Consumers. The values in tables 37 and 38 are from Watt and Merrill (1963), and are the averages of published and unpublished data, in most cases, taken from similar tabular data published by the USDA, universities, and agricultural experiment stations. The values in both tables show the composition of grapefruit in raw, processed, and prepared forms. The values in table 37 are presented in terms of the amounts of nutrients for 100 grams of edible portion of the product, and in table 38, the values are presented in terms of the amount of nutrient in the edible portion of 1 pound of product as purchased. Both tables include data for energy, proximate composition, 5 vitamins, and the minerals calcium, phosphorus, iron, sodium, and potassium. The water content, ash, and fiber have been included in table 37 but not in table 38. In table 38, the column headed "Refuse" represents the average amounts of material removed and discarded in the preparation of food; the rest is the edible portion, including only the part or parts that most individuals in the United States eat. Table 38 is of special interest to the average purchaser, since he can get a better idea of the amounts of food components when he purchases by weight. These values may also be adapted for use in evaluating grapefruit in institutional and national food supplies.

Results from Other Countries. Chace *et al.* (1904) were among the earliest to determine the chemical composition of citrus juices. Chace and his coworkers tested the juice of Cuban (Toronja) grapefruit that had been sent to Washington, D.C. The juice contained from 9.9 to 13.3 per cent solids.

López-Domínguez and Cady (1921), in Puerto Rico, picked and tested fruits at biweekly intervals between September, 1916, and January, 1917. The average soluble-solids values for the 3 varieties tested were: Duncan, 8.7 per cent; Marsh, 7.9 per cent; and Triumph, 9.92 per cent. They reported the following changes as occurring in grapefruit during the process of maturation:

1. Percentage of juice increases as the season advances.
2. Peel thickness tends to remain constant from September to February.
3. In general, soluble solids also remain constant during this period.
4. Acids noticeably decrease during the test period, which shows that most of the acids are produced at the beginning of the season.
5. The soluble solids:acid ratio of 7:1 was reached before the fruits were fully colored. The green color predominated in some fruits that had a ratio of 8:1 or 9:1.
6. Sucrose increased until the ratio was 7:1, while the invert sugar increased continually.

Juritz (1925), in Pretoria, South Africa, was another of the early workers to become interested in determining the composition of citrus fruits. In the reference cited, his results pertained mostly to oranges, but he gave the following values for grapefruit juice: density, 0.73; peel, 32.3 per cent; volume of juice, 182 ml; soluble solids, 8.5 per cent; acids, 1.45 per cent; pH, 3.4; and total sugars per 100 ml of juice, 5.47 grams, of which 0.86 gram was sucrose and 4.61 grams reducing sugars.

In their studies on the composition of Trinidad grapefruit, Hardy and Rodríguez (1935) concluded that in comparison with foreign fruit, the proportions of juice and pulp were somewhat higher in local fruit, and that the proportions of peel and albedo were correspondingly lower. Their data on the composition of grapefruit from 2 localities in Trinidad with contrasting soil types are given in table 58 of this chapter.

Joachim and Panditteseker (1939) obtained the values shown in table 39 for grapefruit of several varieties grown in a number of localities in Ceylon. The percentage of juice varied from 30.5 to 55.2; the solids:acid ratio, from 4.6 to 8.9. The average percentage of soluble solids was 8.5 in 13 samples of Marsh grapefruit from 8 localities, and 8.7 per cent from 2 Cecily, 1 Ellen, 1 McCarty, 1 Foster, 1 Triumph, and 4 Walters samples from 5 localities. These authors imported and determined the composition of grapefruit from South Australia, South Africa, and California. When they compared these values, together with published results from other countries, they concluded that the composition of most samples of their local grapefruit was in every respect equal to, and in some cases superior to, that of grapefruit grown in other countries.

Hall (1943), in New South Wales, determined the effect of rootstock and of area where grown on the specific gravity of freshly picked whole Marsh grapefruit, and on juice percentage, soluble-solids and acids contents, and soluble solids:acids ratio. His results are given in table 139, chapter 4.

Ellis and Tai (1949) determined the composition of the juice of grapefruit from different parishes in Jamaica. The soluble-solids values for all 90 groves tested were: maximum, 11.7 per cent; minimum, 9.7 per cent; over-all average, 10.7 per cent. They concluded that grove variation in a given district was greater than variations between groves in different districts. Their data on juice percentages are given in table 150 of chapter 4. See also, page 356.

Sugars

As a group, sugars constitute over half of the soluble solids in grapefruit juice. Several sugars are present.

Widdowson and McCance (1935), stressing the importance of knowing the kinds and amounts of carbohydrates in a given food, determined several of the most important digestible carbohydrates in the edible portion of the grapefruit, and obtained the following percentages: glucose, 1.95; fructose, 1.24; sucrose, 2.14; starch, 0; total carbohydrates, 5.33; also, water, 90.7. These values are the averages of 4 to 6 samples from each of several different localities (see their table 3).

In testing for sugars in filtered, single-strength grapefruit juice, McCready *et al.* (1950) found 8 per cent soluble solids, 4.36 per cent reducing sugars as

[*Turn to page 75.*]

TABLE 37

Composition of the Edible Portions of Grapefruit, Fresh or Processed.*

Edible portions, fresh or processed	Water (per cent)	Food energy (cal)	Protein (gr)	Fat (gr)	Carbohydrates		Ash (gr)	Calcium (mg)	Phosphorus (mg)	Iron (mg)	Sodium (mg)	Potassium (mg)	Vitamin A values (IU)	Thiamin (mg)	Riboflavin (mg)	Niacin (mg)	Ascorbic acid (mg)
					Total (gr)	Fiber (gr)											
Raw Fruit																	
Pulp—pink, red, white																	
All varieties	88.4†	41	0.5	0.1	10.6	0.2	0.4	16	16	0.4	1	135	80	0.04	0.02	0.2	38‖
California and Arizona (Marsh Seedless)	87.5	44	.5	.1	11.5	.2	.4	32	20	.4	1	135	10	.04	.02	.2	40‖
Florida, all varieties	89.1	38	.5	.1	9.9	.2	.4	15	15	.4	1	135	80	.04	.02	.2	37‖
Texas, all varieties	87.7	43	.5	.1	11.3	.2	.4	15	15	.4	1	135	★§	.04	.02	.2	38
Pulp—pink, red																	
Seeded (Foster Pink)	88.6	40	.5	.1	10.4	.2	.4	16	16	.4	1	135	440	.04	.02	.2	39‖
Seedless (including Marsh Pink, Redblush)	88.6	40	.5	.1	10.4	.2	.4	16	16	.4	1	135	440	.04	.02	.2	36‖
Pulp—white																	
Seeded (Duncan; other varieties)	88.2	41	.5	.1	10.8	.2	.4	16	16	.4	1	135	10	.04	.02	.2	38‖
Seedless (Marsh Seedless)	88.9	39	.5	.1	10.1	.2	.4	16	16	.4	1	135	10	.04	.02	.2	37‖
Juice—pink, red, white																	
All varieties	90.0	39	.5	.1	9.2	tr	.2	9	15	.2	1	162	80	.04	.02	.2	38‖
California and Arizona (Marsh Seedless)	89.0	42	.4	.1	10.2	tr	.3	9	15	.2	1	162	10	.04	.02	.2	40‖
Florida, all varieties	90.4	37	.5	.1	8.8	tr	.2	9	15	.2	1	162	80	.04	.02	.2	37‖
Texas, all varieties	89.2	42	.5	.1	10.0	tr	.2	9	15	.2	1	162	★	.04	.02	.2	38
Juice—pink, red																	
Seeded (Foster Pink)	90.0	38	.5	.1	9.1	tr	.3	9	15	.2	1	162	440	.04	.02	.2	39‖
Seedless (including Marsh Pink, Redblush)	90.0	39	.4	.1	9.3	tr	.2	9	15	.2	1	162	440	.04	.02	.2	36‖
Juice—white																	
Seeded (Duncan; other varieties)	89.6	40	.5	.1	9.5	tr	.3	9	15	.2	1	162	10	.04	.02	.2	38‖
Seedless (Marsh Seedless)	90.2	38	.5	.1	9.0	tr	.2	9	15	.2	1	162	10	.04	.02	.2	37‖
Canned Products																	
Segments, solids and liquid																	
Water pack, with or without artificial sweetener	91.3	30	.6	.1	7.6	.2	.4	13	14	.3	4	144	10	.03	.02	.2	30
Sirup pack	81.1	70	.6	.1	17.8	.2	.4	13	14	.3	1	135	10	.03	.02	.2	30
Juice																	
Unsweetened	89.2	41	.5	.1	9.8	tr	.4	8	14	.4	1	162	10	.03	.02	.2	34
Sweetened	86.2	53	.5	.1	12.8	tr	.4	8	14	.4	1	162	10	.03	.02	.2	31

Frozen concentrated juice—unsweetened																	
Undiluted	62.0	145	1.9	.4	34.6	.1	1.1	34	60	.4	4	604	30	.14	.06	.7	138
Diluted with 3 parts water, by volume	89.3	41	.5	.1	9.8	tr	.3	10	17	.1	1	170	10	.04	.02	.2	39
Frozen concentrated juice—sweetened																	
Undiluted	57.0	165	1.6	.3	40.2	.1	.9	28	50	.3	3	508	20	.12	.05	.6	116
Diluted with 3 parts water, by volume	87.8	47	.4	.1	11.4	tr	.3	8	14	.1	1	144	10	.03	.01	.2	33
Dehydrated juice (crystals)																	
In dry form	1.0	378	4.8	1.0	90.3	.4	2.9	87	155	1.0	10	1,572	80	.36	.16	1.7	350
Prepared with water (1 pound yields about 1 gallon)¶	89.5	40	.5	.1	9.6	tr	.3	9	16	.1	1	167	10	.04	.02	.2	37
Blended juice—grapefruit and orange, canned																	
Unsweetened	88.7	43	.6	.2	10.1	.1	*.4	10	15	.3	1	184	100	.05	.02	.2	34
Sweetened	86.9	50	.5	.1	12.2	.1	.4	10	15	.3	1	184	100	.05	.02	.2	34
Frozen concentrate, unsweetened (undiluted)	59.1	157	2.1	.5	37.1	.1	1.2	29	47	.4	2	623	380	.23	.03	1.1	144
Frozen concentrate, unsweetened (diluted with 3 parts water, by volume)	88.4	44	.6	.1	10.5	tr	.4	8	13	.1	tr	177	110	.06	.01	.3	41
GRAPEFRUIT PEEL—CANDIED	17.4	316	.4	.3	80.6	2.3	1.3	..‡	..	..	..	...	..	...	...	..	..

*Adapted from Watt and Merrill (1963), their table 1.
†Values given are per 100 grams of substance, and are definitely calculated.
‡Blanks indicate the lack of reliable data for a constituent believed to be present in a measurable amount.
§A star indicates that values are *imputed*, usually from another form of the food or from a similar food. For white-fleshed varieties, the value is about 10 international units (IU) per 100 grams; for red-fleshed, about 440 IU.
‖Values are weighted by monthly and total season shipments for marketing as fresh fruit.
¶One pound equals 0.454 kilogram; one gallon equals 3.785 liters.

TABLE 38

NUTRIENTS IN THE EDIBLE PORTIONS OF 1 POUND (0.454 KILOGRAM) OF GRAPEFRUIT, IN FRESH OR PROCESSED FORMS.*

Edible portions, fresh or processed	Refuse (per cent)	Food energy (cal)	Protein (gr)	Fat (gr)	Carbohydrate total (gr)	Calcium (mg)	Phosphorus (mg)	Iron (mg)	Sodium (mg)	Potassium (mg)	Vitamin A values (IU)	Thiamin (mg)	Riboflavin (mg)	Niacin (mg)	Ascorbic acid (mg)
RAW FRUIT—USED FOR SEGMENTS¶															
Pink, red, white															
All varieties	51†	91	1.1	0.2	23.6	36	36	0.9	2	300	180	0.08	0.04	0.4	84‖
California and Arizona (Marsh Seedless)	57	86	1.0	.2	22.4	62	39	.8	2	263	20	.07	.03	.3	78‖
Florida, all varieties	50	86	1.1	.2	22.5	34	34	.9	2	306	180	.08	.04	.4	84‖
Texas, all varieties	50	98	1.1	.2	25.6	34	34	.9	2	306	★§	.08	.04	.4	85
Pink, red															
Seeded (Foster Pink)	52	87	1.1	.2	22.6	35	35	.9	2	294	960	.08	.03	.4	85‖
Seedless (including Marsh Pink, Redblush)	49	93	1.2	.2	24.1	37	37	.9	2	312	1,020	.09	.04	.4	83‖
White															
Seeded (Duncan; other varieties)	55	84	1.0	.2	22.0	33	33	.8	2	276	20	.08	.03	.4	78‖
Seedless (Marsh Seedless)	51	87	1.1	.2	22.4	36	36	.9	2	300	20	.08	.04	.4	82‖
RAW JUICE AND FRUIT USED FOR JUICE¶															
Pink, red, white (all varieties)															
Chilled juice	0	177	2.3	.5	41.7	41	68	.9	5	735	360	.17	.07	.8	171‖
Fruit used for juice	51	87	1.1	.2	20.5	20	33	.4	2	360	180	.08	.04	.4	84‖
California and Arizona (Marsh Seedless)															
Chilled juice	0	191	1.8	.5	46.3	41	68	.9	5	735	50	.17	.07	.8	181‖
Fruit used for juice	57	82	.8	.2	19.9	18	29	.4	2	316	20	.07	.03	.4	78‖
Florida (all varieties)															
Chilled juice	0	168	2.3	.5	39.9	41	68	.9	5	735	360	.17	.07	.8	169‖
Fruit used for juice	50	84	1.1	.2	20.0	20	34	.5	2	367	180	.08	.04	.4	84‖
Texas (all varieties)															
Chilled juice	0	191	2.3	.5	45.4	41	68	.9	5	735	★	.17	.07	.8	171
Fruit used for juice	50	95	1.1	.2	22.7	20	34	.5	2	367	★	.08	.04	.4	85
Pink, red															
Seeded (Foster Pink)															
Chilled juice	0	172	2.3	.5	41.3	41	68	.9	5	735	2,000	.17	.07	.8	177‖
Fruit used for juice	52	83	1.1	.2	19.8	20	33	.4	2	353	960	.08	.03	.4	85‖
Seedless (including Marsh Pink, Redblush)															
Chilled juice	0	177	1.8	.5	42.2	41	68	.9	5	735	2,000	.17	.07	.8	163‖
Fruit used for juice	49	90	.9	.2	21.5	21	35	.5	2	375	1,020	.09	.04	.4	85‖

White															
Seeded (Duncan; other varieties)															
Chilled juice	0	181	2.3	.5	43.1	41	68	.9	5	735	50	.17	.07	.8	172‖
Fruit used for juice	55	82	1.0	.2	19.4	18	31	.4	2	331	20	.08	.03	.4	78‖
Seedless (Marsh Seedless)															
Chilled juice	0	172	2.3	.5	40.8	41	68	.9	5	735	50	.17	.07	.8	169‖
Fruit used for juice	51	84	1.1	.2	20.0	20	33	.4	2	360	20	.08	.04	.4	83‖
CANNED PRODUCTS															
Segments, solids and liquid															
Water pack, with or without artificial sweetener	0	136	2.7	.5	34.5	59	64	1.4	18	653	50	.13	.08	.9	137
Sirup pack	0	318	2.7	.5	80.7	59	64	1.4	5	612	50	.13	.08	.9	137
Juice															
Unsweetened	0	186	2.3	.5	44.5	36	64	1.8	5	735	50	.13	.07	.8	154
Sweetened	0	240	2.3	.5	58.1	36	64	1.8	5	735	50	.13	.07	.8	141
Frozen concentrated juice—unsweetened	0	658	8.6	1.8	156.9	154	272	1.8	18	2,740	140	.63	.27	3.0	626
Frozen concentrated juice—sweetened	0	748	7.3	1.4	182.3	127	227	1.4	14	2,304	110	.53	.23	2.5	526
Dehydrated juice (1 per cent moisture, crystals)	0	1,715	21.8	4.5	409.6	395	703	4.5	45	7,131	360	1.63	.70	7.9	1,588
Blended juice—grapefruit and orange, canned															
Unsweetened	0	195	2.7	.9	45.8	45	68	1.4	5	835	450	.23	.08	1.0	154
Sweetened	0	227	2.3	.5	55.3	45	68	1.4	5	835	450	.23	.08	1.0	154
Frozen concentrate, unsweetened	0	712	9.5	2.3	168.3	132	213	1.8	9	2,826	1,720	1.03	.14	5.0	653
GRAPEFRUIT PEEL—CANDIED	0	1,433	1.8	1.4	365.6	..‡	..	..	..	...	...	...	...	..	...

*Adapted from Watt and Merrill (1963), their table 2.
†Values given are definitely calculated, and are based on 1 pound of substance.
‡Blanks indicate the lack of reliable data for a constituent believed to be present in a measurable amount.
§A star indicates that values are *imputed*, usually from another form of the food or from a similar food. For white-fleshed varieties, the value is about 20 international units (IU) per pound; for red-fleshed, about 1,000 IU.
‖Values are weighted by monthly and total season shipments for marketing as fresh fruit.
¶Where whole, raw fruit is considered, the following is understood to be included: "refuse; rind, rag, seeds, membranes, handling loss."

TABLE 39

THE EFFECTS OF VARIETY AND LOCALITY ON THE COMPOSITION OF GRAPEFRUIT GROWN IN CEYLON.*

Sugars: acid ratios	Juice (per cent)	Total soluble solids (per cent)	Acids (per cent)	Solids: acid ratios	Reducing sugars (per cent)	Sucrose (per cent)	Total sugars (per cent)	Ascorbic acid (mg/ 100 ml)	pH
4.00†	42.8	8.5	1.48	6.1	3.20	2.28	5.48	41.1	3.5
4.00‡	44.3	8.7	1.28	7.1	2.76	2.24	5.00	37.9	3.7

*From Joachim and Panditteseker (1939).
†Averages of 13 samples of Marsh grapefruit from 8 localities in Ceylon.
‡Averages of 2 Cecily, 1 Ellen, 1 McCarty, 1 Foster, 1 Triumph, and 4 Walters from 5 localities in Ceylon.

TABLE 40

SUGARS IN GRAPEFRUIT JUICE.*

Sources	Sugars, per cent of juice		
	Reducing	Sucrose	Totals
1. Chace *et al.* (1904), Cuban fruit analyzed in Washington, D.C.	....	....	6.63
2. Hawkins and Magness (1920), Florida common	2.95	2.82	5.43
3. Hawkins (1921), Fruit sent from Florida to Washington, D.C.	4.30	2.80	7.25
4. López-Domínguez (1921), Duncan, Puerto Rico	3.03	2.10	5.26
López-Domínguez (1921), Marsh, Puerto Rico	3.08	1.66	4.83
López-Domínguez (1921), Triumph, Puerto Rico	3.05	3.13	6.35
5. Juritz (1925), South African grapefruit	4.61	0.86	5.47
6. Harding and Fisher (1945), Duncan	3.0–5.1	2.4–4.0	5.0–8.3
Harding and Fisher (1945), Marsh	2.3–4.8	2.6–3.1	5.1–7.8
7. Roberts and Gaddum (1937), Florida Marsh	3.44	1.34	4.78
Roberts and Gaddum (1937), Florida seedy variety	3.96	2.24	6.20
8. Joachim and Panditteseker (1939), Ceylon, Marsh, average 8 acres.†	3.20	2.28	5.48
Joachim and Panditteseker (1939), Ceylon, average 6 varieties, 5 acres	2.76	2.24	5.00
9. Nolte *et al.* (1942*a*), Florida press juice from dehydration plant	4.23	2.40	6.63
10. McCready *et al.* (1950), filtered single-strength juice	4.36	....	5.00
11. Sinclair and Bartholomew (1944), mean values for 13 rootstocks, fresh juice, Riverside	5.26	2.55	7.81
12. Imperial Valley of California, near Brawley	4.46	3.61	8.07

*For other sugar values, see various other tables, such as 36, 39, 47.
†One acre equals about 0.405 hectare.

glucose, 5 per cent total sugars, and 0.38 per cent flavanones. By paper chromatography they found that sucrose, glucose, and fructose were the only sugars present, except that trace quantities of acid-hydrolyzable glycosides (flavanones) containing galactose and rhamnose were found.

Most workers have reported the sugars only as reducing (or invert or simple) and sucrose (or disaccharides). The reducing sugars predominate, as shown in table 40, a compilation of the sugar values in grapefruit juice obtained by many workers.

Many investigators have reported that sugars increase and acids decrease as the season advances. Usually before or at maturity, the sucrose decreases and the reducing sugars increase. The same changes occur while the fruit is kept in storage. Sucrose is broken down into glucose and fructose. The glycosides (naringin) may also be broken down into sugars and other substances.

Collison (1913), one of the earliest workers to study the composition of grapefruit juice, reported that the decrease in sucrose and the resulting increase in reducing sugars did not occur until the fruit reached maturity.

On the other hand, Grebinskii (1940), in Russia, reported that the sucrose content of grapefruit juice reached a maximum and then began to decrease long before the fruit was fully mature.

Hilgeman and Smith (1940), in Arizona, made studies on the kinds and amounts of sugars in Marsh grapefruit to determine whether such data would give a reliable indication of fruit maturity (figure 13). Tests were made every 14 days, September through May, with the results listed on page 76.

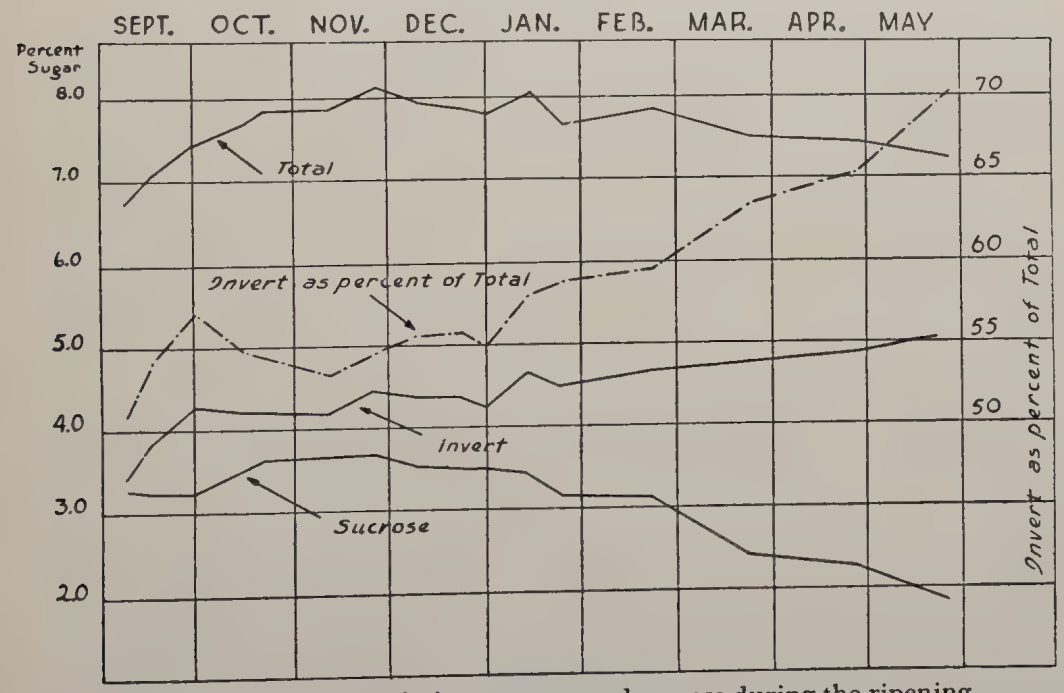


Fig. 13. Changes in invert sugar and sucrose during the ripening of Arizona grapefruit. (From Hilgeman and Smith, 1940.)

1. Changes in the grapefruit, unlike those of some other fruits (pear and date), occurred gradually throughout the growing season.
2. Total sugars increased until midwinter, and then decreased.
3. Invert sugars changed very little during mid-fall, then gradually increased.
4. Sucrose increased rapidly in the early fall, then decreased in late winter and early spring.
5. Fruits having relatively low amounts of invert sugars remained longer on the tree without softening, and underwent less deterioration in commercial storage.
6. Low nitrogen levels in the soil caused an increase in the total amount of sugars, but accelerated the hydrolysis of sucrose. High nitrogen caused the reverse.

Further data on the proportions of reducing sugars and sucrose are given in tables 35, 39, and 40 of this chapter.

Sucrose Synthesis in Citrus Fruits. Bean (1960) reported on the mechanisms of sucrose synthesis in oranges and lemons, using tracer techniques with isolated tissues and with intact small fruit. These 2 categories have widely different sugar-storage patterns, as indicated by the fact that mature lemons contain little or no sucrose in any of their tissues, whereas sucrose is the major storage sugar of the orange. The sugar-storage pattern of grapefruit should be similar to that of the orange.

Labeled glucose-C^{14} or fructose-C^{14} was incorporated into sucrose in isolated peel slices and excised vesicles from lemons of all ages (figure 14). The highest rate of sucrose synthesis occurred in the albedo tissue of the fruit, and the amount formed by the albedo remained surprisingly high throughout the growth period. Percentages of activity in the flavedo and juice vesicles, however, decreased significantly with increase in fruit size.

Bean (1960) also reported that the soluble solids of orange juice contained an active system for the synthesis of sucrose from free fructose and uridine diphosphate glucose (UDPG), but that lemon juice had no such activity in the soluble fraction. The particulate sediment, however, did possess a good capacity for sucrose synthesis. This seems to indicate that the sucrose-synthesizing enzyme in lemons might be soluble protein which is absorbed on the particles during juice processing because of low pH. In fact, lemon protein is usually isolated from the insoluble fraction of lemon juice, and not from the soluble phase of the juice.

Figure 15 shows the results of studies on intact fruits in terms of the specific activities (top graph) of the sugars found in the various fruit tissues, and the ratios (bottom graph) of activities of monosaccharide components of sucrose synthesized from glucose-C^{14} and fructose-C^{14} in tissue preparations of intact lemon and orange fruits. In the intact fruit, glucose is incorporated into sucrose much more rapidly than fructose. Using glucose as a substrate, sucrose attained the highest specific activity, as shown by the fact that relatively low activities remained in the monosaccharides (top graph). When glucose-C^{14} was the substrate, the free fructose pool also became labeled in both lemons

and oranges. When labeled fructose was imbibed, it was converted into sucrose at a much reduced rate in both fruits. The lemon flavedo was the only tissue of both fruits in which the specific activity of sucrose exceeded that of fructose. It should be pointed out that when labeled fructose is fed, a small amount of label appears in the free glucose of lemons, but no conversion to free glucose appears in the orange tissue.

The data in figure 15 (bottom graph) show that activity was present in both components of sucrose, irrespectively of which labeled monosaccharide was used as a substrate. In most experiments, the monosaccharide moiety corresponding to the sugar fed retained a somewhat higher value of labeling. The ratios that approach unity indicate that a rapid state of equilibrium

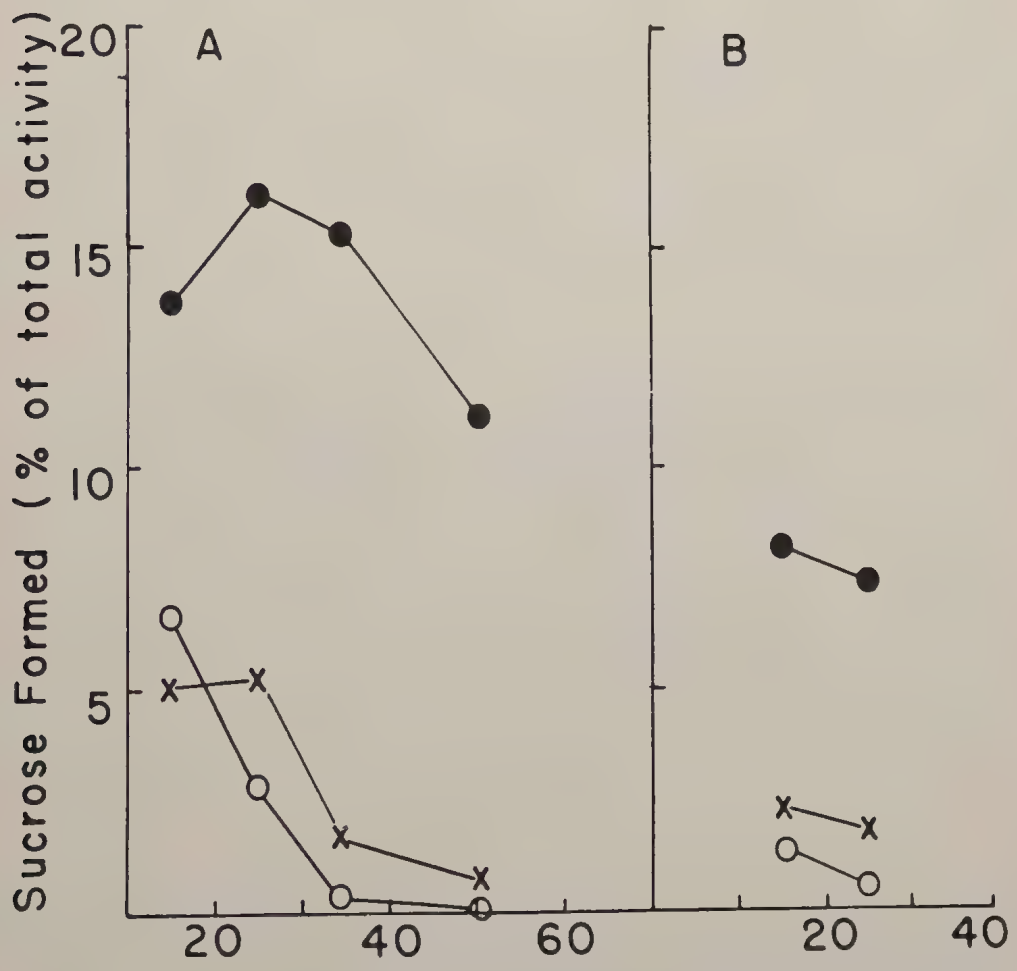
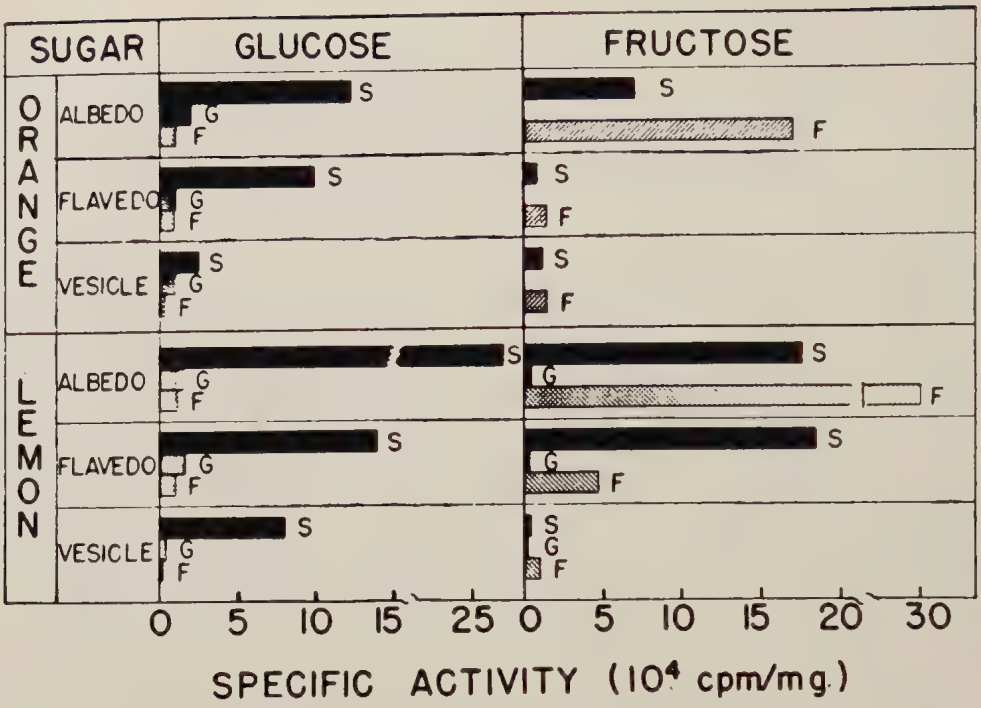


Fig. 14. *A:* Formation of sucrose from glucose in isolated tissues from lemons of various ages. *B:* Formation of sucrose from fructose in tissue preparations from lemons of 2 ages. Black ovals indicate sucrose formed in albedo tissue slices; open ovals, sucrose formed in flavedo tissue slices; X's, sucrose formed in excised vesicles. No activity was found in free fructose with glucose–C^{14} as substrate or in free glucose when fructose–C^{14} was used. Total free fructose was higher than free glucose in the smallest fruits, but was approximately equal in larger fruits. (From Bean, 1960.) Fruit diameters are shown in millimeters.



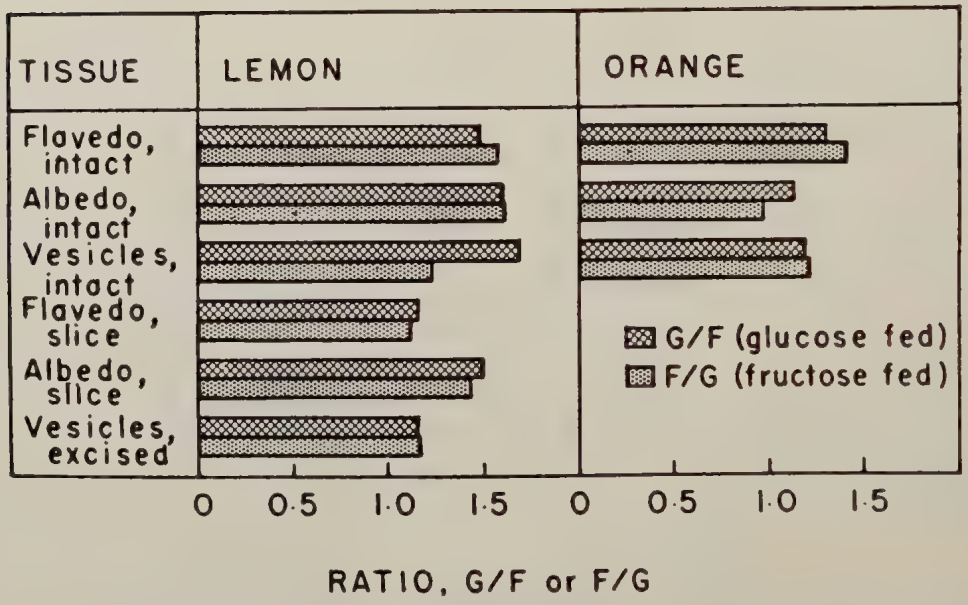


Fig. 15. *Top:* Specific activity of sugars in the different tissues of intact lemons and oranges after uptake of glucose–C^{14} or fructose–C^{14}. Solid bars indicate specific activity of sucrose; cross-hatched bars, specific activity of fructose; speckled bars, specific activity of glucose. *Bottom:* Ratios of activities of monosaccharide components of sucrose synthesized from glucose–C^{14} and fructose–C^{14} in tissue preparations of intact lemon and orange fruits. Cross-hatched bars indicate ratio of activity of glucose to fructose from tissues given glucose–C^{14}; speckled bars, ratio of activity of fructose to glucose in tissues given fructose–C^{14}. Control experiments in which the ratios were determined after giving uniformly labeled sucrose indicated variability of 5 per cent in final ratios. (From Bean, 1960.)

occurred between both monosaccharides in the tissues. In the sucrose component isolated from different orange tissues, the fructose to glucose activity ratio in sucrose of the albedo was 1:0, of the flavedo 1:4, and of the juice vesicles 1:2. These ratios were obtained on tissues from the same experiment, indicating that some dilution of the glucose portion of the sucrose may have occurred after the hexoses left the albedo, or that independent synthesis took place in the other tissues. When glucose was fed, the activity ratio of glucose to fructose was 1:1 in the albedo and 1:3 and 1:2 in the flavedo and juice sacs, respectively, indicating that changes occurred in those tissues.

Acids

TWO MEASURES of acidity are used in physiological studies on grapefruit: pH value and titratable acidity. In modern biochemical and biological research, the hydrogen ion activity (concentration of free or dissociated hydrogen ions) is usually expressed in terms of the pH scale. Pure water at 25° C. (77° F.) has a pH of 7.0 or a hydrogen ion concentration of 1.00×10^{-7} M. The pH of water or of an aqueous solution is related to the hydrogen ion activity by the following equations:

$$pH = -\log [H^+],$$

or

$$[H^+] = 10^{-pH} = \text{antilog } (-pH).$$

The titratable acidity (often called "total free acidity" or "total" or "free" acids) of a substance is the total amount of hydrogen ions that can react with a base; i.e., the free hydrogen ions plus those that will become free during the process of titration. Equal volumes of 0.1 *N* hydrochloric acid and 0.1 *N* acetic acid have the same total acidity, but they are markedly different in hydrogen ion activity. Because these 2 acids have different dissociation constants, it follows that they have different pH values.

Grapefruit contains a relatively high concentration of organic acids. The titratable acidity may constitute 8 to 15 per cent of the total soluble solids of the juice from the edible portion. The titratable acidity of the juice is highly associated with the quality of the fruit. In the standard maturity test (ratio of soluble solids to acids discussed in the following section), the titratable acids, as anhydrous citric, are determined by titrating a known quantity of juice, usually 10 ml, with 0.1 *N* sodium hydroxide. Consequently, nearly all reports on this subject and most general analyses of grapefruit juice (see the early portion of this chapter) contain numerous data on the titratable acidity of the expressed juice, usually given as percentage by weight of the fresh juice or of the total soluble solids or of the total solids.

Many of the biochemical reactions occurring in the fruit during growth and maturity depend upon the concentrations of the various acid constituents

that compose the titratable acidity. The rates and types of reactions in citrus fruits are highly correlated with the pH, which in turn is directly related to the free and combined acids in the juice. The factors considered collectively as the buffer system of the fruit are important in regulating the proper reactions (pH) in the juice.

Factors Affecting Titratable Acidity. Many investigators have shown that the concentration of titratable acids in grapefruit gradually decreases as the fruit develops and matures (Collison, 1913; Chace and Church, 1924; Hilgeman, 1941; Sinclair and Bartholomew, 1944; Harding and Fisher, 1945). Data showing these changes from Ross (1941) in Florida and from Krezdorn and Cain (1952) in Texas are given in tables 45 and 46, respectively, a little later in this chapter.

The amount of titratable acids in the juice of grapefruit at the time of picking also depends on the rootstock (see table 42) and the climatic environment in which the fruit is grown (table 41).

Changes in pH During Fruit Development. Caldwell (1934) showed that in the early development of grapefruit, the pH of the expressed juice was related to the water content of the fruit. Figure 16 shows graphically, for seedling grapefruit from the June bloom, the changes in pH of whole fruit and of pulp, in total weight, in water content, and percentage increase in weight plotted against time of sampling between July and November. Young grapefruit, for a short period after setting, has pH values approximately equivalent to the vegetative tissues of the tree. During this period of high pH, or low acidity, the fruit has a high solids content. This period is succeeded by a short transition period in which the active acidity of the young fruit rises rapidly from the low values of the vegetative tissues to the high values of the developing fruit. With the initial rise in active acidity (decrease in pH), the fruit absorbs water very rapidly, so that there results a reduction of the solids content and a condition of maximum hydration of the fruit. Caldwell concluded that the period of rapid increase in active acidity and water content was at the period of the most rapid increase in weight and volume of the fruit. Therefore, the 3 curves in figure 16, showing the increase in active acidity, in water content, and in percentage increase in weight, begin to rise almost simultaneously and continue upward together.

Harding and Fisher (1945) made pH determinations on the composited juice of grapefruit samples collected at monthly intervals for 4 seasons, 1939–40 to 1942–43 (figure 17). The first sample collected had a slightly higher pH value than those collected late in September or October. Beginning in January, the pH values increased gradually as the fruit matured. During the 10-month period in which those samples were taken, the range in active acidity that occurred amounted to a pH of 0.3. This relatively small change in pH indicates the strong buffer capacity of grapefruit juice,

[*Turn to page 84.*]

TABLE 41

TOTAL FREE ACIDITY OF GRAPEFRUIT JUICE FROM SAMPLES GROWN IN DIFFERENT REGIONS.

Investigators	States and areas	Varieties	Sampling dates	Titratable acids: per cent of fresh weight of juice
Chace and Church (1924)....	California:			
	San Dimas	Marsh	June 12, 1916	1.59
	Upland		June 14, 1916	1.65
	Riverside		June 1, 1917	1.88
	Corona		June 3, 1917	1.65
	Thermal		February 14, 1916	1.58
	El Centro		February 17, 1917	1.90
Hilgeman (1941).....	Arizona:		December 1–January 15	
	Salt River Valley	Marsh	1935	1.82
			1936	1.61
			1937	1.71
			1938	1.78
			1939	1.85
	Yuma	Marsh	1936	1.41
			1937	1.63
			1938	1.59
			1939	1.62
Sinclair and Bartholomew (1944).....	California:	Mean values of grapefruit from 13 different root-stocks.		
	Riverside		May 19, 1938	1.82
	Brawley		November 8, 1938	1.76
Harding and Fisher (1945)...	Florida	Average total acid, very ripe fruit picked late in season. Marsh and Duncan grapefruit on rough-lemon and sour-orange stocks.	May 6–11	1.00–1.28
Wood and Reed (1938).....	Texas	Marsh Seedless grapefruit on sour orange (10-year-old trees).	November 8–22, 1934	1.56

TABLE 42

ACTIVE ACIDITY (pH) OF GRAPEFRUIT JUICE AS AFFECTED BY SEASON, VARIETY, ROOTSTOCK, AND AREA IN FLORIDA WHERE GROWN.*

Variety	Rootstock	Locality (Florida)	Crop seasons	Aug. 25	Sept. 25–30	Oct. 23–28	Nov. 20–25	Dec. 18–23	Jan. 15–20	Feb. 12–17	March 11–16	April 8–13	May 6–11
Marsh	Rough lemon	Davenport	1939–40	3.13†	3.12	3.05	3.15	3.12	3.15	3.18	3.19	3.26	3.46
			1940–41	3.20	3.10	3.09	3.18	3.17	3.19	3.27	3.28	3.30	3.55
			1941–42	3.12	3.11	3.11	3.04	3.01	3.04	3.16	3.14	3.21	3.38
			1942–43	2.96	2.97	2.95	2.97	2.97	3.08	3.07	3.04	3.10	3.12
Marsh	Rough lemon	Lake Hamilton	1940–41	....	3.10	3.05	3.10	3.10	3.15	3.09	3.17	3.18	3.37
			1941–42	3.11	3.09	3.06	3.05	2.96	3.08	3.10	3.13	3.19	3.26
			1942–43	3.01	3.00	3.00	3.05	3.04	3.03	3.06	3.28	3.36	3.39
Marsh	Rough lemon	Windermere	1939–40	3.07	3.05	3.10	3.07	3.07	3.20	3.17	3.20	3.07	3.32
			1940–41	3.17	3.10	3.10	3.08	3.09	3.16	3.19	3.23	3.21	3.27
Marsh	Rough lemon	Bradenton	1939–40	3.09	3.03	3.08	3.08	3.10	3.05	3.10	3.14	3.22	3.31
			1940–41	3.19	3.22	3.06	3.08	3.08	3.14	3.16	3.22	3.19	3.30
			1941–42	3.08	3.03	3.04	3.07	3.08	3.16	3.13	3.17	3.29	3.31
			1942–43	3.04	3.00	3.00	2.97	2.98	2.98	3.04	3.07	3.10	3.22
Marsh	Rough lemon	Fort Pierce	1939–40	3.13	3.06	3.11	3.15	3.17	3.20	3.23	3.20	3.34	3.38
			1940–41	3.12	3.03	3.07	3.10	3.10	3.12	3.15	3.21	3.27	3.40
			1941–42	3.10	3.12	3.15	3.16	3.17	3.17	3.16	3.17	3.28	3.29
			1942–43	3.05	3.08	2.97	3.02	2.95	2.95	3.02	3.08	3.08	3.07
Marsh	Rough lemon	Homestead	1939–40	3.04	3.08	3.13	3.11	3.16	3.13	3.15	....	....	....
			1940–41	3.17	3.11	3.16	3.22	3.18	3.21	3.27	3.30	3.34	3.47
			1941–42	....	3.04	3.09	3.10	3.10	3.28	....	....	....	....
Marsh	Sour orange	Davenport	1939–40	3.13	3.14	3.17	3.20	3.20	....	....	....	....	....
			1940–41	3.22	3.10	3.15	3.24	3.13	3.26	3.24	3.32	3.30	3.41
			1941–42	3.15	3.11	3.07	3.08	3.08	3.10	3.18	3.20	3.24	3.42
Marsh	Sour orange	Bradenton	1939–40	3.02	3.00	3.05	3.04	3.02	3.03	3.03	3.09	3.11	3.25
			1940–41	3.13	3.15	3.00	3.03	3.00	2.97	3.10	3.19	3.13	3.14
			1941–42	3.13	3.10	3.13	3.02	3.09	3.17	3.21	3.25	3.40	3.48
			1942–43	3.05	2.95	2.92	2.95	2.93	3.00	3.05	3.07	3.08	3.22
Marsh	Sour orange	Vero Beach	1939–40	3.09	3.06	3.07	3.11	3.13	3.14	3.10	3.24	3.20	3.38
			1940–41	3.12	3.04	3.09	3.12	3.16	3.16	3.20	3.20	3.28	3.39
			1941–42	3.14	3.15	3.19	3.15	3.18	3.15	3.16	3.18	3.33	3.39
			1942–43	2.93	2.96	3.03	2.93	2.93	2.95	3.05	3.07	3.12	3.22

			1940–41	3.30	3.24	3.20	3.27	3.22	3.20	3.29	3.28	3.31	3.45
			1941–42	3.25	3.14	3.15	3.12	3.15	3.18	3.20	3.29	3.33	3.49
			1942–43	3.07	3.08	3.04	3.10	3.10	3.11	3.12	3.12	3.16	3.26
Duncan	Rough lemon	Lake Hamilton	1940–41	....	3.21	3.18	3.21	3.20	3.25	3.21	3.27	3.28	3.38
			1941–42	3.16	3.18	3.10	3.13	3.15	3.13	3.18	3.18	3.21	3.29
			1942–43	3.07	2.99	3.05	3.04	3.08	3.05	3.05	3.12	3.12	3.19
Duncan	Rough lemon	Palmetto	1939–40	3.24	3.17	3.17	3.18	3.16	3.20	3.21	3.20	3.28	3.50
			1940–41	3.28	3.18	3.18	3.22	3.21	3.24	3.28	3.31	3.28	3.38
			1941–42	3.15	3.07	3.10	3.12	3.12	3.25	3.27	3.30	3.31	3.38
			1942–43	3.11	3.15	3.11	3.17	3.17	3.19	3.25	3.21	3.29	3.34
Duncan	Rough lemon	Fort Pierce	1939–40	3.19	3.15	3.18	3.20	3.20	3.18	3.20	3.24	3.25	3.34
			1940–41	3.22	3.17	3.12	3.13	3.15	3.12	3.20	3.12	3.24	3.21
			1941–42	3.04	3.04	3.17	3.19	3.19	3.18	3.20	3.20	3.26	3.33
			1942–43	3.05	3.07	3.00	3.07	3.04	2.98	3.07	3.04	3.09	3.18
Duncan	Rough lemon	Homestead	1939–40	3.14	3.12	3.14	3.17	3.19	3.10	3.17	....	....	....
			1940–41	3.25	3.20	3.22	3.23	3.22	3.21	3.22	3.28	3.32	3.42
			1941–42	....	3.14	3.13	3.13	3.13	3.30	....	....	....	....
Duncan	Sour orange	Davenport	1939–40	3.24	3.21	3.25	3.22	3.27	3.31	3.31	3.31	3.39	....
			1940–41	3.30	3.15	3.17	3.24	3.22	3.26	3.31	3.32	3.33	3.43
			1941–42	3.27	3.19	3.08	3.08	3.06	3.16	3.24	3.27	3.31	3.43
Duncan	Sour orange	Palmetto	1939–40	3.14	3.15	3.14	3.15	3.11	3.18	3.21	3.18	3.28	3.46
			1940–41	3.25	3.12	3.13	3.18	3.13	3.18	3.21	3.26	3.26	3.47
			1941–42	3.20	3.09	3.09	3.07	3.03	3.15	3.18	3.19	3.21	3.34
			1942–43	3.22	3.08	2.94	2.98	3.03	3.12	3.12	3.12	3.14	3.16
Duncan	Sour orange	Vero Beach	1939–40	3.13	3.10	3.11	3.11	3.11	3.14	3.11	3.15	3.28	3.32
			1940–41	3.22	3.08	3.15	3.18	3.19	3.20	3.22	3.23	3.29	3.40
			1941–42	3.10	3.11	3.12	3.12	3.15	3.17	3.19	3.21	3.28	3.39
			1942–43	3.04	3.04	3.05	2.98	3.06	3.06	3.08	3.10	3.13	3.32
Foster Pink	Sour orange	Bradenton	1939–40	3.17	3.15	3.10	3.20	3.17	3.21	3.19	3.19	....	....
			1940–41	3.22	3.20	3.12	3.20	3.13	3.07	3.20	3.24	3.25	3.38
Thompson	Sour orange	Bradenton	1941–42	3.19	3.05	3.02	2.98	2.99	3.08	3.09	3.15	....	....
			1942–43	3.03	2.98	2.86	3.03	2.99	3.09	3.15	3.12	3.32	3.50

*Adapted from Harding and Fisher (1945).
†All pH values are the means of duplicate determinations.

as shown by the fact that during the sampling period the titratable acidity decreased considerably with large changes in fruit volume. For more detailed data, see table 42. See also, page 331, chapter 4.

Effects on pH of Variety, Rootstock, and Location. The data of table 42 show the pH of grapefruit juice as influenced by season, variety, rootstock, and the area in Florida in which the fruit was grown, as reported by Harding and Fisher (1945). It can be seen that variation in pH of the juice from fruits collected in different groves was relatively small. These authors stated that the pH of grapefruit juice was influenced more by the variety than by the rootstock. The pH values of the juice of Duncan grapefruit were higher than those of the Marsh. In Marsh, the rootstock had only slight influence on the pH values, but in Duncan, lower pH values were found in fruit from the sour-orange stock than in that from rough lemon.

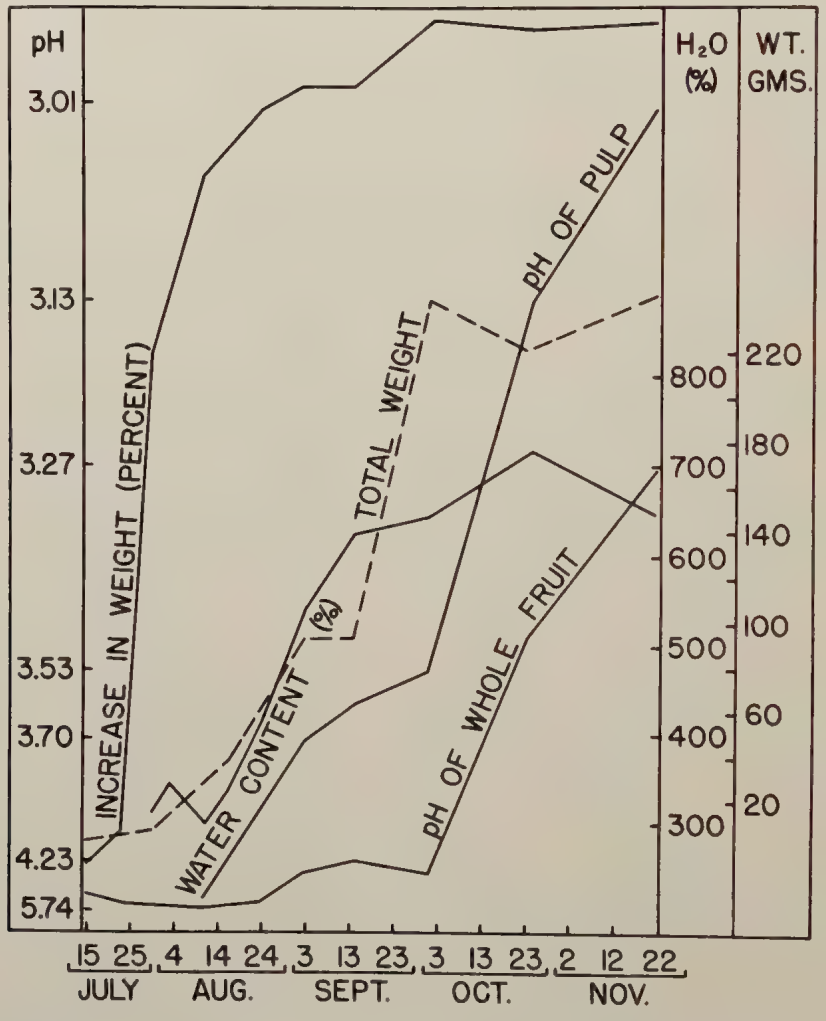


Fig. 16. Changes in pH, water content, total weight, and percentages of increase in weight for seedling grapefruit, June bloom. (From Caldwell, 1934.)

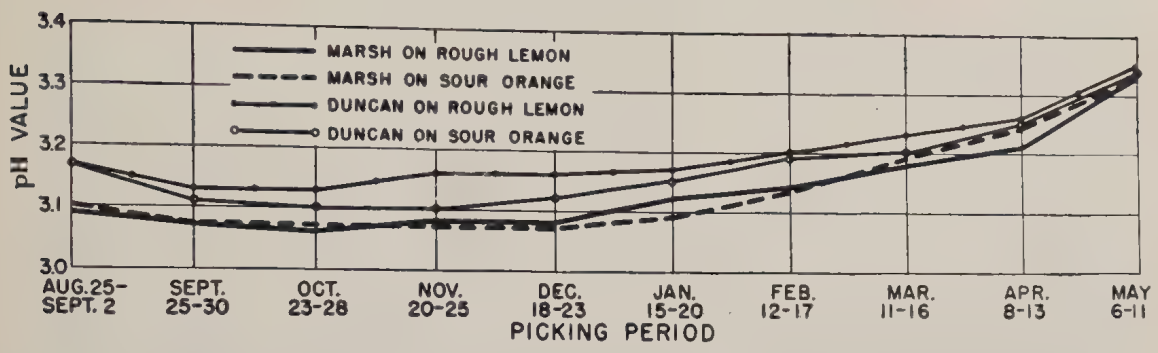


Fig. 17. Seasonal changes in the pH values of Marsh and Duncan grapefruit on rough-lemon and sour-orange rootstocks, 1939–43. (From Harding and Fisher, 1945.)

Variables Affecting the Relation of pH to Titratable Acidity. The pH of grapefruit juice is determined chiefly by the dissociation constant, pK_a, and the ratio of the combined to the free acids of the juice. The following equation is approximate for calculating the pH of weak acids.

$$\text{pH} = pK_a - \log \text{salt/acid},$$

where the term "salt" indicates that of the organic acid present.

Sinclair and Eny (1946*a*) showed that the citric and malic acid contents of grapefruit juice were the chief constituents that contributed to the hydrogen ion concentration. Since the citric and malic acid systems in grapefruit juice (see table 43 and page 88) can be classed as weak acids, this equation may be applied.

Within the changes of concentration that occur in the juice of mature grapefruit, the variation in pK_a is very slight; it may therefore be assumed to be a constant in this discussion. That leaves the factor, log salt/acid, as the only one that may vary the pH.

The dilution effect of the water in the juice during fruit growth is undoubtedly an important variable. The water content may vary considerably, and thus cause variations in the concentration of the acid, although the absolute amount of acid present remains constant. The variations in water content do not change the value of log salt/acid, however, for the salt and acid would be diluted or concentrated in the same proportions. The pH of the juice might therefore remain constant while the concentration of titratable acidity varied over a wide range.

Since differences in water content of grapefruit cause a partial variation in fruit composition, it is possible to eliminate this variable by expressing the free acidity as milliequivalents per gram of soluble solids instead of percentages of fresh weight. Figure 18 shows the relationship between free acidity and pH of the juice within a pH range between 3.1 and 3.6, which represents free acid changes that occur during the last 4 months of fruit development and maturation. In other words, the pH of the juice is a function of the log

of the free acid concentration when calculated as milliequivalents per gram of soluble solids (Kilburn and Davis, 1959).

The opposite may also occur; i.e., the pH may vary, while the acid concentration remains unchanged. This effect would be caused by variation in the value of the factor log salt/acid. If the acid concentration remains constant, then the numerator, salt concentration, must cause the pH variation. It is probable that fruit from different trees and groves will vary sufficiently in the amount of salt in the juice to produce a significant variation in pH.

With a system such as grapefruit juice, a combination of variables probably produces the wide variation in the relation of pH to the percentage of titratable acidity on a fresh-weight basis. For example, dilution could result in a decrease in concentration of the acid and, at the same time, a change in the absolute amount of salt might decrease the pH. Other factors probably enter into the relation, but the 2 variables just discussed are considered the most important.

Relation of pH to Combined Acid (Salt). Sinclair and Eny (1946*a*, *b*) determined the pH–salt relations of grapefruit juice by comparing the titration curve of the juice with that of a pure citric acid solution (figure 19).

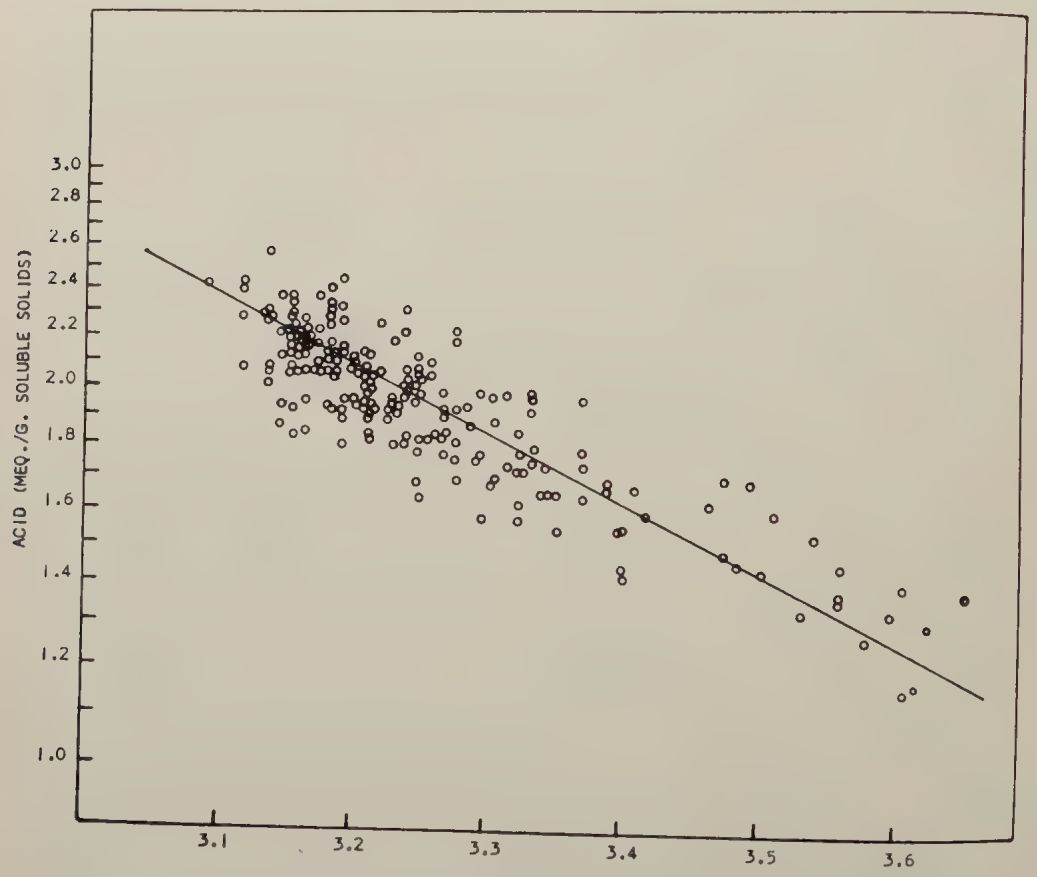


Fig. 18. Relationships between acid content and pH in grapefruit juice. (From Kilburn and Davis, 1959.)

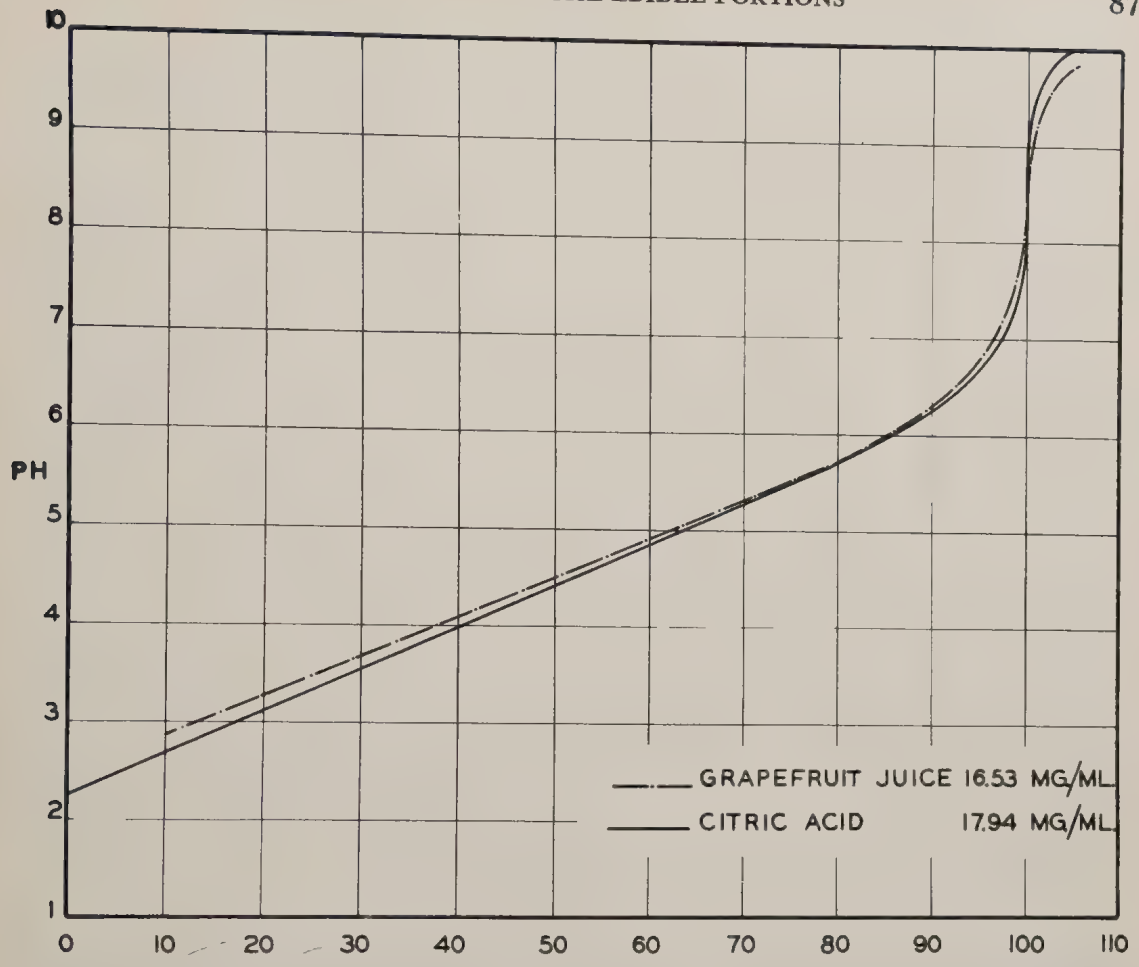


Fig. 19. Titration curves of grapefruit juice and pure citric acid solution, showing relationship of pH to salt content. The initial point on the grapefruit-juice curve was corrected for salt occurring normally in it. Lower figures show percentage of acid neutralized. (From Sinclair and Eny, 1946*a*.)

The solution used in securing the data for the citric acid curve contained 17.94 mg of citric acid per ml; the titratable acid (as anhydrous citric) in the grapefruit juice was 16.53 mg per ml. The pure citric acid solution had a pH of 2.25; the grapefruit juice had an initial pH of 2.87. The initial point on the grapefruit juice curve is located on the graph at pH 2.87, which corresponds to a combined acid, or salt, content of 10 per cent. The percentage of acid neutralized for all points other than the initial one included the combined acid present in the original sample and the sodium citrate formed by the neutralization of part of the free acid. The 2 curves are nearly parallel, and the pH values are very close, those of grapefruit juice being slightly higher than those of citric acid for a given amount of combined acid. Grapefruit juice titrates like a pure citric acid solution because the free acid in the juice is composed chiefly of citric acid (see "Specific Organic Acids Present" on page 89), and the ratio of free acid:combined acid is great enough to diminish the salt effect on the pH. The quantity of cations that is avail-

able in the juice for combining with the organic acids is relatively small in comparison with the concentration of free acids. To plot an accurate titration curve of grapefruit juice, a correction must be made for the combined acid naturally occurring in the juice. Grapefruit juice, like all weak acids, is most effective in its buffer capacity at a pH approximating the dissociation constant of the buffer acid (pK_a).

Kilburn and Davis (1959) determined the relationship between the salt content of various grapefruit juices and their respective pH values (figure 20). The mean values of the salt content appeared to be the same within the pH range 3.1 to 3.6. This situation indicates that the average salt content of grapefruit juice, reported as milliequivalents per gram of soluble solids, remains nearly constant throughout the growth and maturation season. It follows, then, that changes in the pH of grapefruit juice are highly correlated with differences in concentration of free acids.

Base-Forming Elements in Grapefruit Juice. Sinclair and Eny (1946*a*, *b*) used the alkalinity of the ash as a measure of the combined acids and the base-forming elements of grapefruit juice (table 43). During the ashing of the juice, the organic materials were burned off, leaving the equivalent mineral elements (cations) as carbonates, oxides, and some sulfates and phosphates. The specific cations combined with the organic acids were not determined.

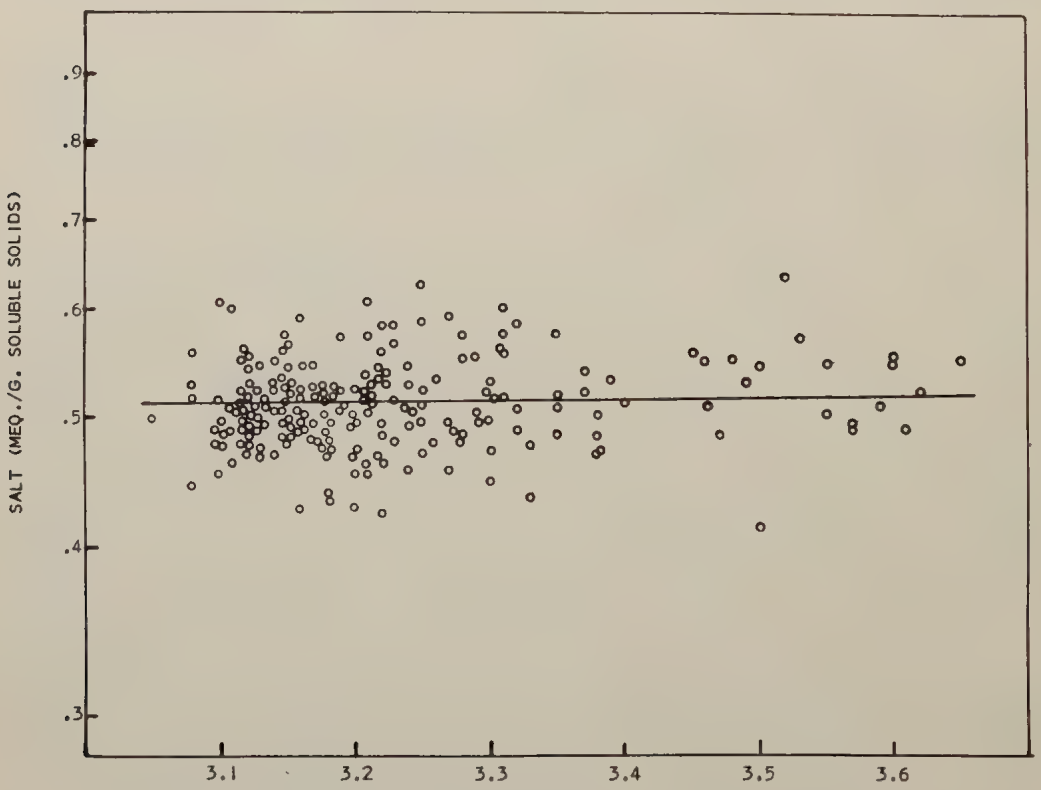


Fig. 20. The relationship between salt content and pH in grapefruit juice. (From Kilburn and Davis, 1959.)

It is highly probable, however, that the citrates were present in the juice as potassium acid citrates, since the potassium concentration accounted for 60 to 70 per cent of the total cations.

Specific Organic Acids Present. Menchikovsky and Popper (1932) determined the organic acids of grapefruit juice, and reported that the total acidity was composed of 98.72 per cent citric acid, 0.05 per cent tartaric acid, 1 per cent malic acid, and 0.23 per cent oxalic acid.

In another test by Sinclair and Eny (1946*a*), the organic polybasic acids of grapefruit juice were precipitated in 80 per cent alcohol with lead acetate. An analysis of this lead-free precipitate gave the organic acids and acid salts naturally present in the juice. The completeness with which lead acetate precipitated the organic acids is evident from the close agreement of the total acid values with those reported for the sum of the citric and malic acids (table 43). The total acid values (as citric) represent all the acid groups precipitated from the juice with lead acetate. That inorganic acid salts were not present in the juice in appreciable amounts is shown by the fact that the citric and malic acids account for all the acid groups in the precipitate.

Clements (1964*a, b*) determined the organic acids in the juice and peel of mature grapefruit (table 44). His results were interesting in that the juice and peel samples were freeze-dried, and the organic acids were subsequently determined by silicic acid chromatography. Cation-exchange resin in the acid form was used to acidify the peel before extraction of the acids with methanol. In agreement with numerous published results, citric acid was found to predominate in grapefruit juice, and malate occurred in low but significant concentrations. Oxalate predominated in the peel, and malonate existed in measurable concentrations. The values reported by Clements for oxalate, citrate, and malate in peels of orange, lemon, and grapefruit agreed closely with those reported by Sinclair and Eny (1947). Biale and Young (1962) found malonic acid in lemon peel. Clements found that, in many instances, the malonic acid content of citrus peel was exceeded only by that of oxalate. He found that citric, malic, oxalic, and malonic acids accounted for 30 to 50 per cent of the total anion in the peel fractions. The paper chromatographic analysis of the effluents showed that at least 15 peaks appeared with each peel, indicating other undetermined components.

Soluble Solids:Acids Ratios

THE EDIBLE QUALITY of grapefruit depends in large measure upon the ratio of sugars to acids in the juice. There is fairly close correlation between total soluble solids and sugar content because a high proportion of the soluble solids are sugars. Because of this, and because the concentration of soluble solids is quickly and simply determined by Brix floating hydrometer or a

[*Turn to page 93.*]

TABLE 43

Analyses of the Organic Acids of Grapefruit Juice.*

Sample number and locality (California)	Sampling date	Stage of fruit maturity	Fruit diameter (cm)	True soluble solids† (per cent)	pH	Free acid (as citric) determined by phenolphthalein titration	
						(mg/ml)	(meq/ml)
Fruit Samples from Packing House							
1. Riverside	July 17	Yellow	8.5	12.6	2.75	20.88	0.326
2. Riverside	July 19	Yellow	8.5	12.5	2.80	21.51	.336
3. Riverside	July 23	Yellow	8.5	10.7	2.86	18.22	.285
4. Sunnymead	July 26	Yellow	10.0	10.3	2.80	18.48	.289
5. Hemet	July 27	Yellow	10.5	10.3	2.86	17.72	.277
6. Riverside	July 31	Yellow	10.5	11.0	2.86	17.53	.274
7. Riverside	Aug. 3	Yellow	8.0	10.8	2.87	17.34	.271
8. West Riverside	Aug. 7	Yellow	10.0	10.8	2.87	18.22	.285
Fruit Samples of Different Stages of Maturity							
9. Riverside	Aug. 20	Green	5.0	7.6	3.30	19.89	.311
10. Riverside	Aug. 24	Green	6.0	7.9	3.08	22.72	.355
11. Riverside	Aug. 26	Green	6.0	7.7	3.08	19.06	.298
12. Indio	Aug. 28	Green	7.5	10.5	2.96	22.84	.357
13. Indio	Aug. 29	Green	8.5	9.8	3.06	19.88	.311
14. Indio	Aug. 31	Green	9.3	9.9	3.08	22.84	.357

[*Continued on the facing page, where the last 11 columns of figures contain additional data.*]

Sample number and locality (California)	Sampling date	Stage of fruit maturity	Acid constituents precipitated by lead acetate								Combined acid (as citric) determined from alkalinity of ash		Free acid
			Total acid titrated (as citric)		Citric acid (pentabrom-acetone method)		Malic acid		Total acid (citric plus malic)				
			(mg/ml)	(meq/ml)	(mg/ml)	(meq/ml)	(mg/ml)	(meq/ml)	(mg/ml)	(meq/ml)	(mg/ml)	(meq/ml)	(per cent)
Fruit Samples from Packing House													
1. Riverside.....	July 17	Yellow	26.58	0.415	22.58	0.353	4.00	0.060	26.44	0.413	1.96	0.031	91.41
2. Riverside.....	July 19	Yellow	26.58	.415	23.58	.368	3.30	.049	26.70	.417	2.08	.032	91.18
3. Riverside.....	July 23	Yellow	24.55	.383	22.94	.358	1.80	.027	24.65	.385	1.90	.030	90.55
4. Sunnymead.....	July 26	Yellow	24.90	.389	23.48	.367	1.62	.024	25.03	.391	2.02	.032	90.14
5. Hemet.....	July 27	Yellow	24.23	.378	21.95	.343	2.45	.037	24.33	.380	2.04	.032	89.68
6. Riverside.....	July 31	Yellow	23.44	.366	21.32	.333	2.30	.034	23.50	.367	1.96	.031	89.94
7. Riverside.....	Aug. 3	Yellow	23.28	.364	20.27	.317	3.46	.052	23.62	.369	1.71	.027	91.02
8. West Riverside...	Aug. 7	Yellow	24.04	.376	22.58	.353	1.80	.027	24.33	.380	1.96	.031	90.29
Fruit Samples of Different Stages of Maturity													
9. Riverside.....	Aug. 20	Green	27.78	.434	25.17	.393	2.85	.043	27.91	.436	4.99	.078	80.00
10. Riverside.....	Aug. 24	Green	29.02	.453	26.43	.413	2.55	.038	28.87	.451	4.49	.070	83.50
11. Riverside.....	Aug. 26	Green	25.93	.405	23.21	.363	2.82	.042	25.93	.405	3.96	.062	82.79
12. Indio.....	Aug. 28	Green	29.03	.453	25.90	.405	2.97	.044	28.74	.449	3.03	.047	88.29
13. Indio.....	Aug. 29	Green	27.51	.430	25.35	.396	2.35	.035	27.59	.431	3.53	.055	84.92
14. Indio.....	Aug. 31	Green	30.29	.473	26.08	.407	4.10	.061	29.96	.468	3.28	.051	87.44

*From Sinclair and Eny (1946*a*).
†Because of the relatively high acid content of the juice, a correction was made on the hydrometer readings to give the true percentage of soluble solids (Stevens and Baier, 1939).

TABLE 44

Organic Acids of the Juice and Peel of Grapefruit from Various Localities.*

PART I

Citric and Malic Acid Contents of Grapefruit Juice

Varieties	Solids (per cent)	Acid (as citric) (per cent)	Solids: acid ratios	pH	Meq per 100 mg solids		Grams per 100 ml juice	
					Malic acid	Citric acid	Malic acid	Citric acid
California (Marsh)	10.1	1.80	5.6	2.9	9	276	0.06	1.79
Arizona	11.6	1.98	5.9	3.1	5	284	.04	2.10
Texas (pink)	9.3	1.14	8.2	3.3	9	201	.06	1.19

PART II

Organic Acids in Grapefruit Peel

Varieties	Weights (grams) (average per fruit)				Meq per gram dry weight of peel				
	Fruit	Pulp	Peel		Total anion extd.	Citric acid	Malic acid	Oxalic acid	Malonic acid
			Wet	Dry					
California (Marsh)	286.2	193.3	90.5	20.3	0.32	0.01	0.03	0.06	0.02
Arizona	361.1	243.7	112.8	28.3	.59	.03	.10	.12	.02
Texas (pink)	395.4	311.7	80.2	18.8	.53	.01	.08	.08	.02

*Adapted from Clements (1964*a*, *b*).

refractometer, the ratio of soluble solids to titratable acids—the maturity ratio, Brix:acids ratio, or soluble solids:acids ratio, calculated by dividing the percentage of soluble solids by the percentage of titratable acids—is the most widely used criterion of maturity in determining the proper time to harvest (see "Maturity Standards" in chapter 4). Soluble solids are also frequently used in analysis to determine such points as varietal differences or the effects of climate, as shown in many of the tables presented in this chapter and in the 2 following ones.

Ratios in Fresh Juice. Collison (1913) concluded that there was a gradual increase in sugars and a decrease in acid concentration as the season advanced. Apparently, all the acid was formed early in the season, but the sugars increased until the fruit was mature.

From 1915 to 1917, Chace and Church (1924) made about 500 determinations on the composition of the juice of Marsh grapefruit growing in many areas of California and, for comparison, some analyses of Arizona and Florida fruit. The period over which the analyses were made began long before and continued long after the stage of maximum maturity. Those who are especially interested in such data are referred to their paper; the tabular data are too extensive to be included here.

Caldwell (1934) determined, about once a week between July 30 and November 11, the solids, both soluble and insoluble, in juice from grapefruit grown in Florida. The changes during this period, in fruit from the June bloom, are shown here.

	July 30	*November 11*
Fruit diameter (centimeters)	1.7–2.0	9.0–9.5
Soluble solids (per cent)	13.28	9.52
Insoluble solids (per cent)	11.14	3.88
Total solids (per cent)	24.42	13.40

Percentages were computed on the basis of fresh weight of the whole fruit. Note the differences in solids in very small fruit as compared with larger sizes. Fruit from February bloom gave similar results.

In many tests on the composition of grapefruit juice, the number of samples and the number of trees and localities from which the fruit was taken have been too few to give reliable results. Ross (1941) avoided such possible criticism by taking his samples from 160-gallon (605-liter) tanks each day as the juice was being prepared for canning. It thus came from many trees and localities in Florida. His values showing seasonal changes in solids, acids, and ratios, and also in ascorbic acid, are presented in table 45. Each value was the average of 10 samples.

Krezdorn and Cain (1952) planned a 5-year test on some of the factors that influenced the production and composition of grapefruit in the Rio Grande Valley of Texas. However, because of the severe freeze in January, 1949, they

were able to carry it on for only 5 months of 1 season, and therefore pointed out that their data were not entirely accurate. Furthermore, it is probable that some off-bloom fruit was included. The differences may not be significant, or may be owing to factors other than variety. Nevertheless, some of their findings are of interest; these are reported in table 46 and in the listings shown below. The varieties tested were Marsh, Thompson, and Webb (Redblush). The Webb trees were 8 to 12 years old; the others, 15 to 20 years. There was a plot of each variety in each of 4 localities, 20 trees in each plot. Fruit sizes ranged from 9 cm at the first picking (August) to nearly 10 cm at the last (December). (See also, table 33.)

1. During this experimental period, the total soluble solids did not increase significantly as the season progressed. The differences were not great, but the average values for Webb were generally lower than those of the others.

2. Titratable acids decreased more than the soluble solids increased as the season advanced (table 46). Variety had an influence on acid values. Thompson was lower than Marsh, and Webb was lower than Thompson. As the season progressed, the magnitude of the differences became greater.

3. The ratios rose rapidly as the season advanced. The table shows that Thompson had higher mean values than Marsh, and Webb was higher than either of the others.

4. The percentage of juice by weight increased as the season advanced (see Krezdorn and Cain's table 2), then leveled off in November and December. Variety had no appreciable effect on the percentage of juice, as shown by the values in table 33, taken from the last half of the authors' table.

Lime *et al.* (1954) reported that in the Ruby (Ruby Red) grapefruit, the average weight increased approximately 2.5-fold, from 207 to 543 grams, during the sampling period, while the titratable acidity decreased from 2.43 to 1.02 per cent (see table 84 of this chapter). It is of interest that the relative

TABLE 45

SEASONAL CHANGES IN SOLUBLE SOLIDS, ACIDS, AND RATIOS OF SOLUBLE SOLIDS TO ACIDS FOR FLORIDA GRAPEFRUIT.*

Month	Ascorbic acid (mg/ml)		Soluble solids (per cent)		Acids (per cent)		Ratios of solids:acids	
	1938–39	1939–40	1938–39	1939–40	1938–39	1939–40	1938–39	1939–40
December	0.44	0.43	10.3	10.0	1.54	1.39	6.7	7.2
January	.46	.44	10.5	10.5	1.49	1.53	7.1	6.9
February	.47	.44	10.7	10.0	1.54	1.39	7.0	7.2
March	.46	.40	10.8	9.8	1.54	1.31	7.2	7.5
April	.45	.40	10.3	9.5	1.55	1.23	7.3	7.7
May	.43	.40	10.2	9.7	1.43	1.26	7.8	7.7
June	.43	.43	10.6	9.8	1.25	1.25	8.5	7.9
Averages	.45	.42	10.8	9.9	1.47	1.34	7.4	7.4

*Adapted from Ross (1941), his table 3.

TABLE 46

EFFECTS OF VARIETY ON SOLIDS, ACIDS, AND THEIR RATIOS, AS EVIDENCED BY THE MEAN VALUES OF 4 LOCATIONS.*

Dates, 1950	Soluble solids (per cent)			Acids (per cent)			Ratios		
	Marsh	Thompson	Webb (Redblush)	Marsh	Thompson	Webb (Redblush)	Marsh	Thompson	Webb (Redblush)
August 21	11.65	11.50	11.33	1.99	1.95	1.82	5.86	5.92	6.29
September 18	11.72	11.46	11.11	1.81	1.74	1.60	6.47	6.58	7.02
October 16	11.29	11.14	11.29	1.68	1.52	1.38	6.94	7.56	8.42
November 13	11.19	11.02	10.87	1.54	1.41	1.26	7.30	7.80	8.85
December 11	11.47	11.39	11.01	1.42	1.31	1.11	8.02	8.67	10.32

*Adapted from Krezdorn and Cain (1952). (Refer also to table 33.)

increase in fruit weight was approximately the reciprocal of the decrease in titratable acidity expressed as a percentage of the fresh weight of the juice. These facts lead to the conclusion that, although the concentration of the titratable acids decreased, the absolute amount of acids calculated on a per-fruit basis showed no such decrease during the approximately 9-month sampling period. To illustrate this fact, the first sample contained 5.03 grams of titratable acid per fruit, and the last sample contained 5.43 grams per fruit. The changes in the Brix:acid ratio were caused by a decrease in acid concentration rather than by an increase in sugar concentration.

It is important for processors and those who concentrate citrus juices to use fruit with as high a soluble-solids content as possible. In their study of the Brix content and Brix:acid ratios of grapefruit used by Florida processors over a 5-season period, Stenstrom and Westbrook (1956) noted that the maximum soluble solids of the juices were attained in March of each of the years studied. After March, the degrees Brix had a slight tendency to decrease, while the Brix:acid ratios continued to increase (figure 21). The increases in ratios were caused by the decrease in concentration of the free acidity as the fruit remained on the trees. Extending from February through June, a large volume of fruit which met the 9.5 per cent soluble solids with minimum

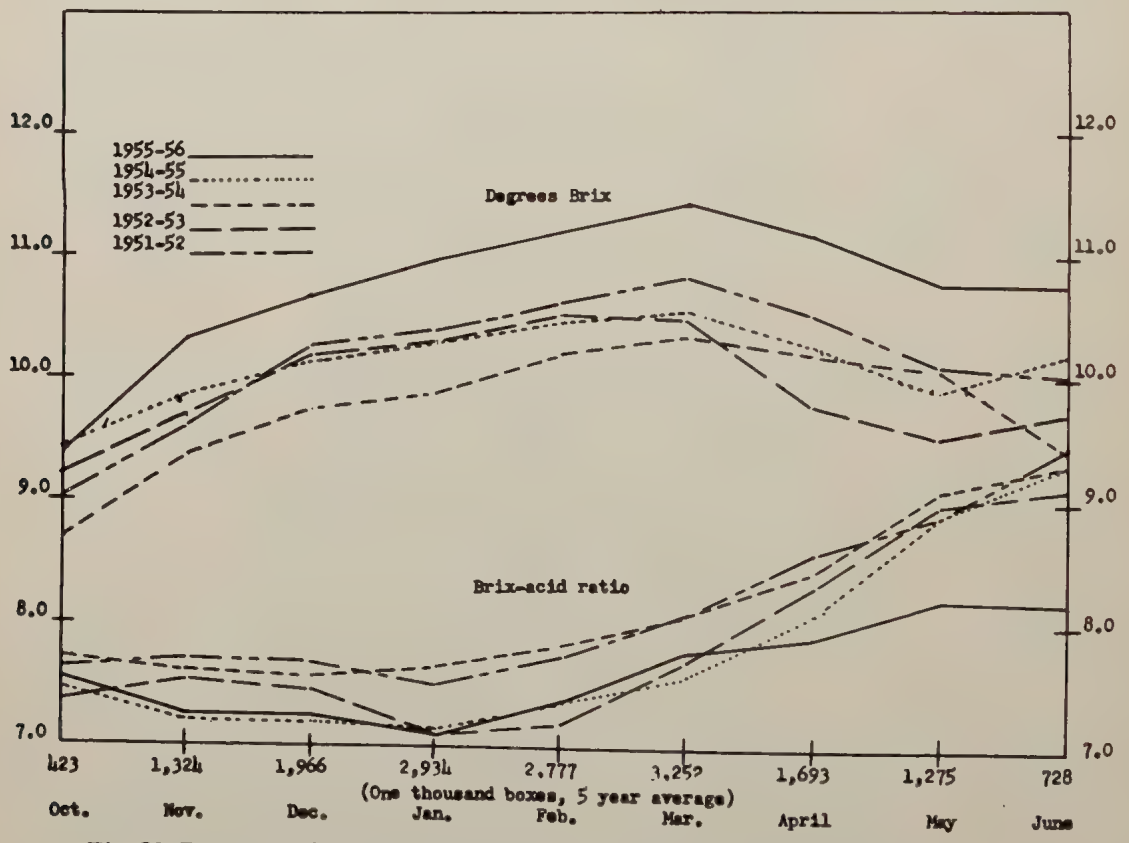


Fig. 21. Degrees Brix and Brix:acid ratios of grapefruit utilized by Florida citrus processors for 5 seasons. (From Stenstrom and Westbrook, 1956.)

ratios of 7.5 to 1 was available to the processor. Without any unusual changes in weather conditions or cultural practices, including insecticidal treatments, the data in the figure indicated to the processor the quality of fruit received at the plant, as demonstrated by the progressive changes that occurred in the Brix and Brix:acid ratios of grapefruit during the 9 months of experimental research.

In Canned Juice. In his tests for ascorbic acid in canned grapefruit juice, Lamb (1946) also determined the degrees Brix of the canned juice immediately after cooling. The values were: for 3 California canneries, 10.9 to 12.1°, averaging 11.3°; and for 4 Arizona canneries, 10.7 to 12.2°, averaging 11.7°.

Juice Gradients. Hilgeman (1941), Baier and Higby (1931*a, b*), and others pointed out that complete extraction of all the juice from the edible portion of the fruit would be necessary for accurate determination of the solids:acid ratio because the composition of mature fruits is not the same in all parts.

Sutherst (1911) and Chace and Church (1930) appear to have been the first to show that there is a higher concentration of soluble solids in the stylar-end pulp than in the stem-end pulp of citrus fruits (oranges). As indicated in the following text, several other workers have shown this to be true for mature fruits, but not necessarily for immature ones.

Baker (1932) found that the juice from the central part of the pulp of grapefruit was higher in acids and soluble solids than that from the outer part. He also found stem-to-stylar gradients in grapefruit, similar to the findings described in the remainder of this section.

Taking the average of samples for 2 dates, Wood and Reed (1938) found that the juice from the stylar half of the fruit was higher in soluble solids, in solids:acid ratio, and in pH, and lower in titratable acidity, than the juice from the stem half. Table 47 presents their data on these points, and also on some other components and characteristics of the 2 halves.

Bartholomew and Sinclair (1941) tested the gradients of a large number of navel and Valencia oranges and a few grapefruit. The results for grapefruit (table 48), as for oranges, indicated that in the pulp of immature citrus fruit the concentration of soluble solids may sometimes be as high in the stem end as in the stylar end, or higher, They found also that among grapefruit picked from the outside of the tree, 4 to 5 feet aboveground, 89 per cent had a higher total soluble-solids content in 3 north-oriented segments than in 3 south-oriented segments, whether the fruits were borne on the north, south, east, or west side of the tree.

Hilgeman (1941) found that titratable acidity was lower and soluble-solids: acid ratio higher near the peel than at the center (table 49), that titratable acidity was lower at the stylar end than at the stem end, and highest near the center of the stem end. The table also shows that soluble solids were higher near the peel in immature fruit, but near the center in mature fruit.

TABLE 47

CHEMICAL COMPOSITION OF JUICE FROM STEM AND STYLAR ENDS OF GRAPEFRUIT.*

(Plat Y—untreated, 10 years old, 1934.)

Fruit half and date	Taste†	pH	Buffer indexes	Soluble solids (degrees Brix)	Percentage composition, wet basis			
					Citric acid (anhydrous)	Invert sugar	Sucrose	Total sugars
Stem end								
Nov. 8	SS–S, VSB	3.20	1.013	11.00	1.70	4.20	2.66	6.86
Nov. 22	A–T, NB	3.15	1.140	10.95	1.64	4.20	2.52	6.72
Averages	T–SS, VSB–NB	3.18	1.076	10.98	1.67	4.20	2.59	6.79
Stylar end								
Nov. 8	SS–S, VSB	3.20	1.013	11.00	1.49	4.48	2.76	7.24
Nov. 22	SS, NB	3.20	1.073	11.05	1.44	4.30	2.76	7.06
Averages	SS–S, VSB–NB	3.20	1.043	11.02	1.46	4.39	2.76	7.15
Both								
Averages	SS–S, VSB–NB	3.19	1.058	11.00	1.56	4.30	2.68	6.90

Fruit half and date	Ratios					Percentage composition, dry basis			
	Brix to acid	Invert sugar to acid	Sucrose to acid	Total sugars to acid	Invert sugars to sucrose	Citric acid (anhydrous)	Invert sugar	Sucrose	Total sugars
Stem end									
Nov. 8	6.47	2.47	1.56	4.04	1.58	15.45	38.18	24.18	62.36
Nov. 22	6.68	2.56	1.54	4.10	1.63	14.98	38.35	26.48	64.83
Averages	6.57	2.51	1.55	4.06	1.62	15.22	38.26	25.33	63.60
Stylar end									
Nov. 8	7.38	3.01	1.85	4.86	1.62	13.54	40.73	25.09	65.82
Nov. 22	7.67	2.99	1.92	4.90	1.56	13.03	38.91	24.98	63.89
Averages	7.55	3.01	1.89	4.90	1.59	13.28	39.82	25.04	64.86
Both									
Averages	7.05	2.76	1.72	4.42	1.60	14.25	39.04	25.18	64.22

*Adapted from Wood and Reed (1938).

†A equals acid; T equals tart; SS equals slightly sweet; S equals sweet; SB equals slightly bitter; VSB equals very slightly bitter; NB equals nonbitter.

TABLE 48

DISTRIBUTION OF SOLUBLE SOLIDS IN STEM AND STYLAR HALVES OF GRAPEFRUIT IN DIFFERENT STAGES OF MATURITY.*

Age of fruit (months)	Concentrations of soluble solids (per cent)		
	Higher in stem end	Higher in stylar end	Equal in both ends
8.5	9	78	13
9.5	0	97	3

*Adapted from Bartholomew and Sinclair (1941).

TABLE 49

VARIATIONS IN THE COMPOSITION OF JUICE FROM INNER AND OUTER PORTIONS OF THE FRUIT.*

October 30, 1938				April 18, 1939			
Fractions of juice	Brix	Acids (per cent)	Brix:acids ratios	Fractions of juice	Brix	Acids (per cent)	Brix:acids ratios
Inside				Inside			
32 per cent	11.38	2.19	5.20	41 per cent	10.15	1.55	6.38
Middle				Middle			
28 per cent	11.87	1.82	6.52	23 per cent	9.85	1.25	7.88
Outside				Outside			
40 per cent	11.95	1.48	8.07	36 per cent	9.63	1.12	8.60

*From Hilgeman (1941).

Ascorbic Acid (Vitamin C)

THE NUTRITIONAL VALUE of grapefruit juice is closely related to its vitamin C content at the time of its consumption, as stated by Wagner *et al.* (1945). Consequently, a great deal of work has been done on this constituent, and many vitamin C values have been published in scientific literature. Table 50 gives a list of vitamin C values found in grapefruit by many authors, as affected by variety, part of fruit, where grown, processed or fresh, and other factors. Hamner (1945) said it was fortunate that the findings at that time appeared to show that the treatments which produced the largest crop yield also gave the maximum yield of vitamins. Booher *et al.* (1942) gave an extensive table on vitamin values of food in relation to processing and other variants. In a review of the literature dealing with the isolation and identification of vitamin C, King (1936) cited 126 research articles, practically all of which were published during the 4-year period, 1932–36. He stated that excellent reviews of earlier work done on vitamin C had been given by Hess (1920), Sherman and Smith (1931), and Waugh (1934). Except for some of

TABLE 50

VITAMIN C CONTENT OF GRAPEFRUIT.

Investigators	Part of fruit, variety, and treatment (if given)	Amounts of vitamin C
Beacham and Bonney (1937)	Juice, Marsh, on sour orange (Florida)	0.38 mg/ml (mean values)
	Juice, Florida common, on rough lemon	0.37 mg/ml (mean values)
Daniel and Rutherford (1937)	Juice, Tresca, shaddock group (Florida)	0.64 mg/ml
	Juice, Davis	0.38 mg/ml
	Juice, Davis	0.34 mg/ml
	Juice, Bowen	0.35 mg/ml
	Juice, Foster Pink	0.33 mg/ml
American Medical Association (1938).	Juice, canned (several brands)	Average 37, range 29–42 mg/100 ml
Hill (1938)	Pulp, fresh (Australia)	33 mg/100 grams
American Medical Association (1939).	Juice, canned, sweetened (commercial brands)	36 mg/100 ml
	Juice, canned, unsweetened	35.2 mg/100 ml
Floyd and Fraps (1939)	Pulp, fresh, Canner's Prolific (Texas)	36.8 mg/100 grams
	Pulp, fresh, Duncan (Texas)	37.0 mg/100 grams
	Pulp, fresh, Foster Pink	44.1 mg/100 grams
	Pulp, fresh, Marsh Seedless	31.3 mg/100 grams
	Pulp, fresh, McCarty	31.3 mg/100 grams
	Pulp, fresh, Thompson	34.7 mg/100 grams
Metcalfe *et al.* (1940)	Juice, fresh, Rio Grande Valley, Texas, November (Marsh)	39.2 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, November (Marsh Pink)	35.5 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, November (Redblush)	36.6 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, November (Marsh Seedless)	35.0 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, November (Foster Pink)	44.9 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, November (Duncan)	41.3 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, February (Marsh)	34.7 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, February (Marsh Pink)	33.5 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, February (Redblush)	31.8 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, February (Marsh Seedless)	34.0 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, February (Foster Pink)	32.3 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, February (Duncan)	36.4 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, April (Marsh)	27.9 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, April (Marsh Pink)	28.5 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, April (Redblush)	24.9 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, April (Marsh Seedless)	30.0 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, April (Foster Pink)	29.5 mg/100 ml
	Juice, fresh, Rio Grande Valley, Texas, April (Duncan)	33.0 mg/100 ml
French and Abbott (1940)	Juice, fresh, Florida common (central)	47 mg/100 ml
		38 mg/100 ml
		46 mg/100 ml
		56 mg/100 ml
	Juice, fresh, Florida (east coast)	44 mg/100 ml
		40 mg/100 ml
		38 mg/100 ml
		41 mg/100 ml
		45 mg/100 ml
		48 mg/100 ml
	Juice, stored 1 month, Florida (central)	54 mg/100 ml

TABLE 50—*Continued*

VITAMIN C CONTENT OF GRAPEFRUIT.

Investigators	Part of fruit, variety, and treatment (if given)	Amounts of vitamin C
Newman and Fellers (1940)	Juice and pulp in tin containers	43–54 mg/100 grams
	Juice in glass containers	39 mg/100 grams
Holmes *et al.* (1941)	Juice, fresh (commercial samples)	48 mg/100 ml (average)
	Juice, canned (commercial samples)	31 mg/100 ml (average)
Cowart (1942)	Juice, no MgO (Duncan)	39.6 mg/100 ml
	Juice, no MgO (Marsh)	35.0 mg/100 ml
	2 per cent MgO (Duncan)	42.7 mg/100 ml
	2 per cent MgO (Marsh)	36.8 mg/100 ml
	4 per cent MgO (Duncan)	45.6 mg/100 ml
	4 per cent MgO (Marsh)	37.1 mg/100 ml
Floyd and Fraps (1942)	Juice, fresh, Marsh (average of 21 samples, Rio Grande Valley, Texas)	41.2 mg/100 grams
	Juice, canned, Marsh (average of 109 samples, Rio Grande Valley, Texas)	33.7 mg/100 grams
Harding and Thomas (1942)	Juice, fresh, Marsh, Duncan, Foster Pink (Florida)	0.40 mg/ml (average)
Hernández (1943)	Juice, fresh (Cuba)	35.3 mg/100 ml
Harding (1944)	Juice, fresh (Florida)	32–62 mg/100 ml
Jones *et al.* (1944*b*, 1945)	Juice, fresh, low N nutrition (Arizona)	0.47 mg/ml
	Juice, fresh, high N nutrition (Arizona)	0.38 mg/ml
Quinones *et al.* (1944)	Juice, fresh	39 mg/100 ml
	Juice, canned	44 mg/100 ml
Ribeiro (1944)	Juice, fresh (Brazil)	57 mg/100 ml
Smith *et al.* (1944)	Peel, size 54 fruits per box	142 mg/100 grams
Anon. (1945)	Juice (Buenos Aires)	48.9 mg/100 grams
Munsell (1945)	Juice, fresh, 14 samples (Puerto Rico)	35.6–52.9 mg/100 ml
Ajon (1946)	Juice (Italy)	59.6 mg/100 grams
Anon. (1947)	Juice, 183 samples (Victoria)	40.6 mg/100 ml (average)
	Juice, 131 samples (New South Wales)	39.6 mg/100 ml (average)
	Juice, 21 samples (Queensland)	40.4 mg/100 ml (average)
	Juice, 29 samples (South Australia)	39.1 mg/100 ml (average)
Potter (1947)	Juice, Marsh (Australia)	30–35 mg/100 ml
Sale (1947)	Juice, 3 to 5 samples (Florida)	41.1 mg/100 ml (average)
	Juice, 3 to 5 samples (Texas)	40.0 mg/100 ml (average)
	Juice, 3 to 5 samples (California–Arizona)	43.5 mg/100 ml (average)
French and Abbott (1948)	Pulp and juice (Florida)	42 mg/100 grams (average)
Sites (1947)	Juice, seasonal average, no-Mg plot (Florida)	35.8 mg/100 ml
	Juice, seasonal average, K + Mg plot (Florida)	41.7 mg/100 ml
Moore (1949)	Juice, after 18 months at 76° F. (24° C.) (bottles)	47 per cent retention
	Juice, after 18 months at 76° F. (24° C.) (cans)	58.4 per cent retention
	Juice, after 18 months at 40° F. (4.4° C.) (bottles)	86.9 per cent retention
	Juice, after 18 months at 40° F. (4.4° C.) (cans)	94.2 per cent retention
Miller and Jablonski (1949)	Seeds, ungerminated (fresh weight)	1.69 mg/100 grams
	Seeds, ungerminated (dry weight)	3.36 mg/100 grams
	Seeds, germinated (fresh weight)	17.35 mg/100 grams
	Seeds, germinated (dry weight)	44.80 mg/100 grams
	Seeds in 1 ton (ungerminated)	9.33 grams/ton
	Seeds in 1 ton (germinated)	177.6 grams/ton
Krezdorn and Cain (1952)	Juice, Marsh Seedless, August 21 to December 11 (Texas)	37.4–45.9 mg/100 ml
	Juice, Thompson, same dates (Texas)	35.9–47.2 mg/100 ml
	Juice, Redblush, same dates (Texas)	35.2–47.6 mg/100 ml

the data in table 50, most of the work reviewed in this section was published after King's review.

Grapefruit as a Source of Vitamin C. Cattoni and González (1932) stated that grapefruit juice had a scurvy-preventing potency equal to that of lemon juice, and that its curative value was superior.

Smith (1944) bought canned fruit and vegetable juices on the market in Tucson, Arizona, and tested them at once for their vitamin C contents. In this respect, citrus juices were found to be superior to the 17 other juices tested. Expressed in terms of diet needs, the value found for grapefruit juice was 41.9 milligrams of vitamin C per half cup (4 ounces) of juice. Margaret Smith (1944) compared her findings with the following daily allowances of vitamin C, as recommended by the National Research Council. Amounts are given in milligrams.

Men	
Adults	75
Boys 12–15 years old	90
Boys 16–20 years old	100

Women		Children	
Adults:		Under 1 year	30
Moderatively active	70	1–3 years old	35
Last half of pregnancy	100	4–6 years old	50
While nursing	150	7–9 years old	60
Girls 13–20 years old	80	10–12 years old	75

Smith *et al.* (1944) reported that whole segments contained from 40 to 80 per cent more vitamin C than strained juice from reamed fruit. They also found that the vitamin C content of the juice of grapefruit of a given size might vary from 27 to 36 milligrams per 100 grams.

Lamb (1946) remarked that from the aspects of convenience and economy, the canning of citrus juices was making an excellent source of vitamin C available to the public.

A food survey by French and Abbott (1948) showed that the intake of vitamin C by a large percentage of the population was far below the optimum. They stated that an adult would need about 1,000 international units (50 mg) daily. This would call for a little over 2 ounces of juice containing 0.80 milligram per milliliter of vitamin C.

Elliott and Schuck (1949) found, in 4 out of 5 tests, that humans utilized crystalline vitamin C or that from the grapefruit equally well.

Seasonal Changes. Melas-Joannides (1939), in Greece, reported results which showed that the more mature fruits from a given tree would sometimes contain more vitamin C than the less mature ones.

Metcalfe *et al.* (1940), in the Rio Grande Valley of Texas, concluded that there was a very definite decrease in the vitamin C content of the fruit (6 varieties) by the end of the season; see table 51.

Ugon and Bertullo (1944) and Krezdorn and Cain (1952) stated that the amount of vitamin C in grapefruit juice decreased as the season advanced. On the other hand, in a later paper, Ugon and Bertullo (1945) reported that it increased. Harding (1944) noted that the concentration of vitamin C tended to decrease as the amount of juice increased with advancing maturity, but that the total amount of vitamin C per fruit increased.

The seasonal decrease in concentration of ascorbic acid in grapefruit should be considered in relation to the increase in volume of juice and in size and weight per fruit during growth and maturation (Harding and Fisher, 1945).

Figure 22 shows that ascorbic acid, when expressed as milligrams per milliliter of juice, decreased significantly during the season. Since growth of the fruit is accompanied by a large increase in juice volume and fruit weight, the concentration of ascorbic acid has decreased; there is less ascorbic acid per milliliter of juice. On the other hand, if the ascorbic acid values are reported on the basis of milligrams per fruit, an increase occurs during the growth and development.

Long *et al.* (1957) analyzed 1,320 grapefruit of different sizes for ascorbic acid during the crop seasons 1954–55 and 1956–57, and reported that small fruits of both Marsh and Duncan had higher concentrations, but a lower total amount of ascorbic acid, than larger ones (table 52).

Sunlight and Vitamin C. Light is not essential for the synthesis of vitamin C in plants, and this fact was substantiated by the results of Harris and Ray (1933), who showed that ascorbic acid was formed in seedlings germinated in complete darkness. However, many published experiments have supported the fact that light is an important controlling influence in the synthesis of ascorbic acid. Diurnal fluctuations occur in the ascorbic acid concentrations of plants grown out of doors; the maximum amount in potato leaves was observed at noon by Smith and Gillies (1940). Intermittent exposures of plants to light and darkness, or the shading of plants, resulted in altering the ascorbic acid concentration in their tissues (Hamner *et al.* 1945). In fact, a significant positive correlation was found to exist between the ascorbic acid

TABLE 51

EFFECT OF SEASON ON THE ASCORBIC ACID CONTENT OF 6 VARIETIES OF GRAPEFRUIT.*

(Milligrams of ascorbic acid per 100 milliliters of juice.)

Months	Marsh plain	Marsh Pink	Redblush	Marsh Seedless	Foster Pink	Duncan
November	39.2	35.5	36.6	....	44.9	41.3
February	34.7	33.5	31.8	34.0	32.3	36.4
April	27.9	28.5	24.9	30.0	29.5	33.0

*Adapted from Metcalfe *et al.* (1940).

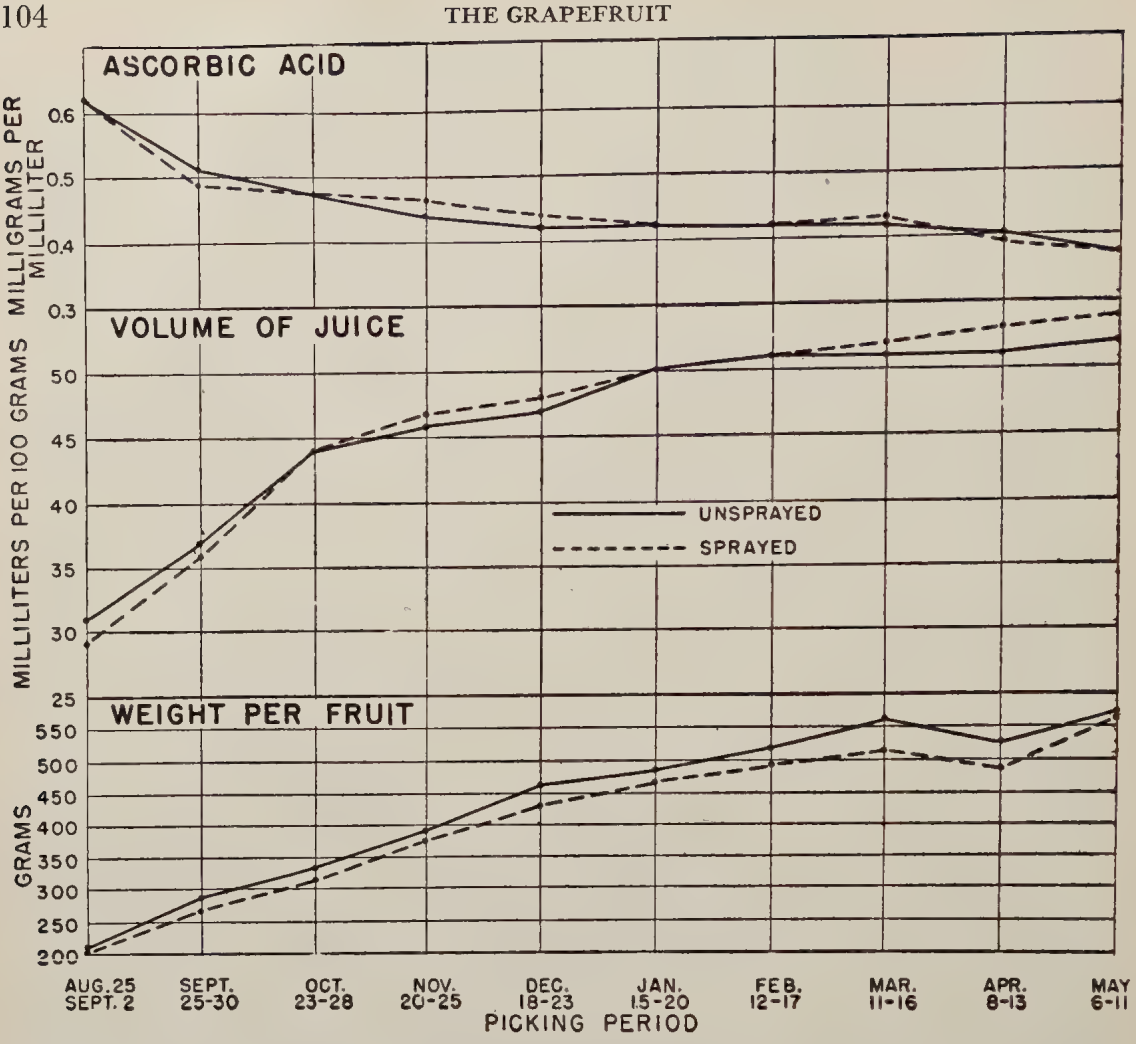


Fig. 22. Relationship of ascorbic acid to volume of juice and weight per fruit of Marsh grapefruit on sour-orange rootstock, when unsprayed, and when sprayed with lead arsenate at different picking periods. (From Harding and Fisher, 1945.)

concentration and light intensity in the leaves of turnip and tomato plants (Somers *et al.*, 1948). Similar data have been reported for fruit at various stages of growth and maturity.

Sah (1936) and Sah and Fang (1937) presented evidence to show that vitamin C would be formed as a result of photosynthesis. More recently, other researchers have stressed the importance of the need for photosynthesis to be in progress in order to supply a sufficient quantity of hexoses from which ascorbic acid is synthesized (Isherwood and Mapson, 1962). Harding *et al.* (1940) and Harding and Fisher (1945) believed that vitamin C was related to the normal process of metabolism, and was not merely a storage constituent of the fruit.

Many workers have reported an effect of light exposure on the vitamin C content. Zilva *et al.* (1935) observed that the red peel of Brawley's seedling apples contained twice as much vitamin C as the green peel. The inference

has been made that the greater quantity of vitamin C in the red peel of the apple was caused by the fact that the red side was the one normally exposed on the tree to direct sunlight. Thus, Harding and Thomas (1942) found that outside fruit had a higher vitamin C content than inside fruit, the difference being significant at the 1 per cent level; and that both Marsh and Duncan grapefruit had a higher vitamin C content in unshaded than in shaded fruit. Jones *et al.* (1945) stated that sunlight tended to increase, and luxuriant growth to decrease, the vitamin C content. In studying many varieties of oranges grown in Florida, Winston and Miller (1948) demonstrated that the vitamin C and total soluble solids of the juice samples were significantly higher in fruit from the outside branches than in that from inside branches of the same tree.

Similarly, Tsushi (1946) and Sites and Reitz (1949) found the largest amount of vitamin C in fruit having maximum exposure to sunlight. Sites and Reitz (1950*b*) also reported that outside fruit grown on the north side of the tree had a lower vitamin C content than fruit from the outer part of the south side. Furthermore, they showed that the total soluble-solids content increased with increase in height on the tree for all classes exposed to light, and that the top outside fruit had the highest total soluble solids. It follows, then, that the vitamin C content of the fruit should show a similar relationship to its height and position on the tree. Figure 23, showing the

TABLE 52

AVERAGE CONCENTRATION AND TOTAL CONTENT OF ASCORBIC ACID IN GRAPEFRUIT OF DIFFERENT SIZES.*

Variety and sizes	Concentrations (mg per 100 ml)	Contents per fruit (mg)
Marsh†		
96	39	71
80	38	81
70	37	86
64	37	93
54	36	104
46	35	114
Duncan‡		
96	40	73
80	39	78
70	38	88
64	38	98
54	37	108
46	34	121

*From Long *et al.* (1957).
†Each value is based on the analysis of 140 fruits.
‡Each value is based on the analysis of 80 fruits.

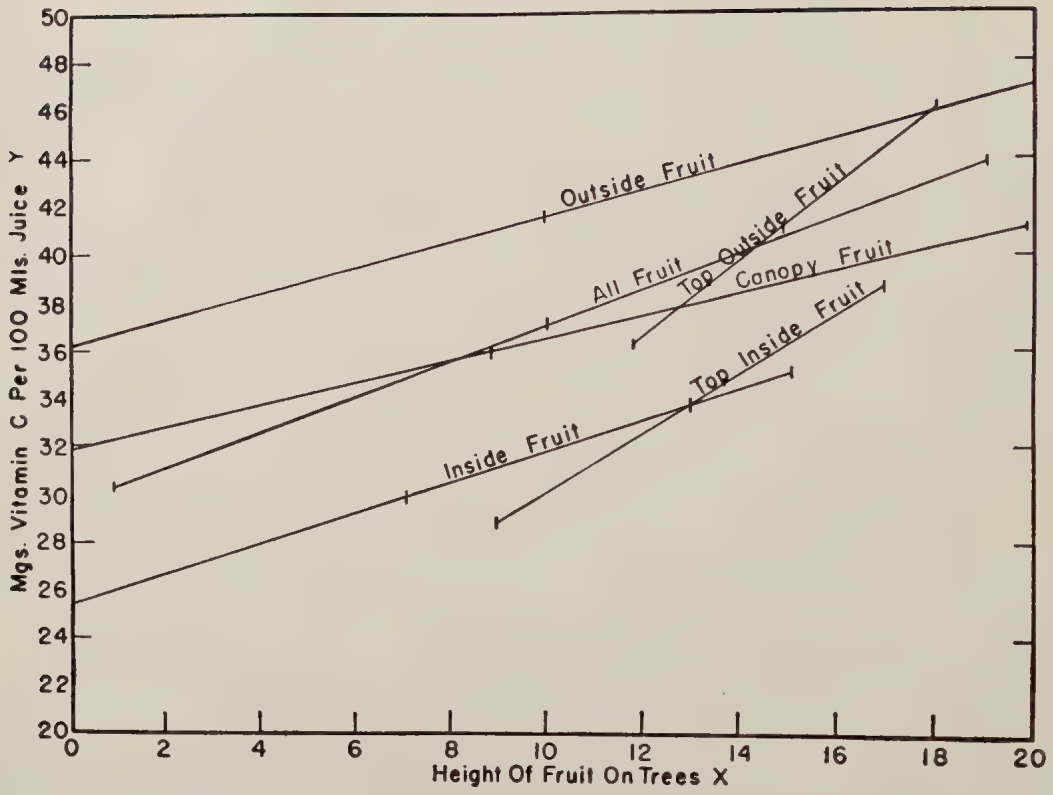


Fig. 23. Regression of vitamin C (milligrams per 100 milliliters of juice) on the height of fruit on trees for all light conditions. (From Sites and Reitz, 1950*b*.)

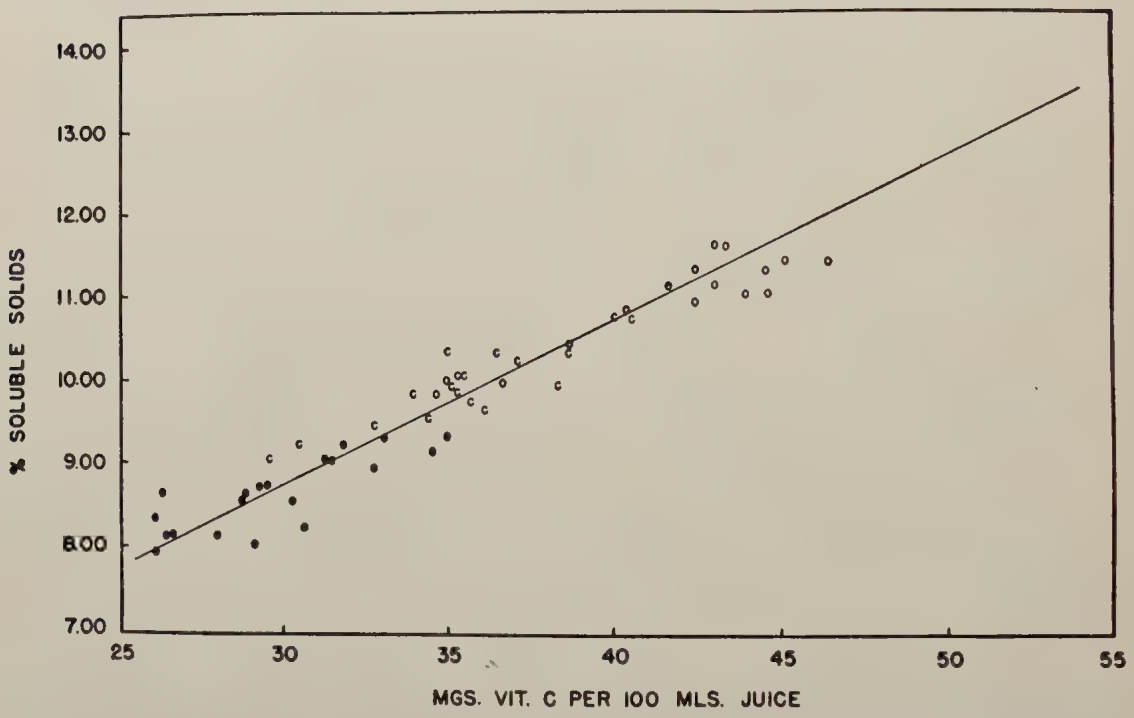


Fig. 24. Regression of soluble-solids values on vitamin C values for fruit in corresponding positions on the tree. Open ovals indicate fruit from the outside portion of the tree; solid ovals, from the inside; and "C" indicates canopy fruit. (From Sites and Reitz, 1950*b*.)

regression of the amount of vitamin C in the juice on the height of fruit for all light classes, indicates that the relationship of height to the vitamin C content is really the effect of light rather than the direct effect of height. If the exposure to light is highly correlated to height of fruit on the tree, the vitamin C content of the fruit would be highly related to either factor—light or height.

These investigators also noted that since the amount of vitamin C and total soluble-solids content are similarly related to the height of fruit on the tree, it follows that a high correlation should exist between vitamin C concentration and the total soluble solids of citrus juices. Figure 24 shows that the correlation coefficient for these two variables was 0.942 for 57 pairs of values.

Fruit color was another factor that was found to be highly correlated with the vitamin C content in the juice (Sites and Reitz, 1950*b*). Figure 25 shows the increase in vitamin C with the development of fruit color from green to orange. It appears from this graph that fruit color is related to light intensity similar to the light conditions that affect total soluble-solids and vitamin C content. In other words, these characteristics—light intensity, total soluble solids, and vitamin C—are significantly correlated with each other, but the primary cause of the relationships is not yet determined.

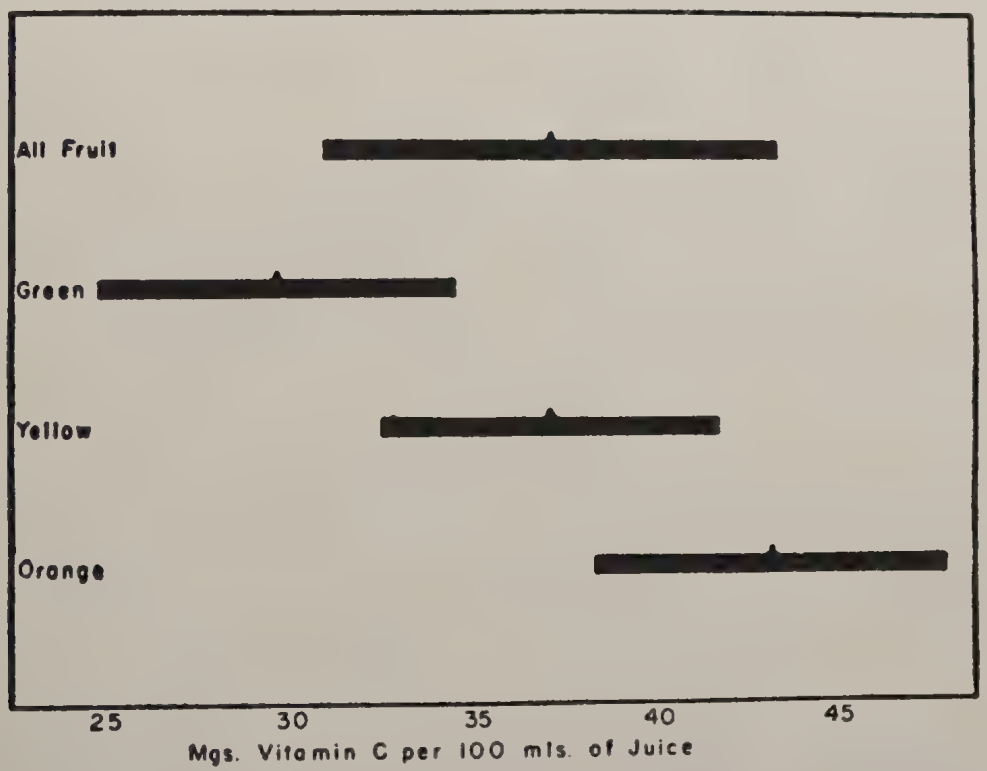


Fig. 25. Vitamin C content of orange juice (milligrams per 100 milliliters) for major rind-color classifications. The length of a bar indicates the mean plus-minus standard deviation, and the point indicates the mean value. (From Sites and Reitz, 1950*b*.)

It should be noted that a considerable body of published data substantiates the fact that temperature has a marked influence on the color change of citrus fruits. Stearns and Young (1942) reported that color change in oranges in the fall did not definitely occur until minimum night temperatures dropped below 55° F. (13° C.), and that the rate of change from green to orange was related to the severity of temperature lowering and the continued occurrences of temperatures below 55° F.

Young and Erickson (1961) placed young Valencia orange trees grafted onto Cleopatra mandarin seedlings in growth chambers, and studied the effects of nighttime-temperature air, daytime-temperature air, and soil temperature on the development of fruit color. All these factors proved to be important in the color changes of the fruit. It was also noted that at temperatures of 20° C. for daytime air, 7° for nighttime air, and 12° for soil (68, 45, and 54° F.), bright orange-colored fruits were produced. A higher level for any of the temperature variables produced fruit with less orange color.

In the foregoing text dealing with the effect of light on vitamin C content, considerable research performed on the orange has been reported, the results of which are believed to be highly applicable to grapefruit under similar experimental conditions. These data are fundamentally important to grapefruit, in that their results bear a direct relationship to the effects of various light intensities on the composition of attached fruits, with particular reference to vitamin C.

Effects of Locality, Variety, and Nitrogen Supply. Metcalfe *et al.* (1940), in the Rio Grande Valley of Texas, tested the effects of variety and grove location on the vitamin C content of 5 different varieties of grapefruit in 6 locations. They concluded that there were rather small differences among the varieties, and considerable but inconsistent variation on the basis of location (table 53). French and Abbott (1940) found no relationship between vitamin C content and grove location, but reported that the fruits from any given

TABLE 53

EFFECT OF VARIETY AND GROVE LOCATION ON VITAMIN C CONTENT.*
(Milligrams per 100 milliliters of juice.)

Locations	Marsh plain	Marsh Pink	Redblush	Foster Pink	Duncan
1	43.8	....	40.7	43.0	....
2	39.0	....	38.0	....	45.3
3	36.0	37.8	....	46.9	38.4
4	40.7	35.8	....	34.8	48.7
5	32.4	35.8	34.8	35.3	36.4
6	43.3	32.6	33.0	35.0	38.0

*Adapted from Metcalfe *et al.* (1940).

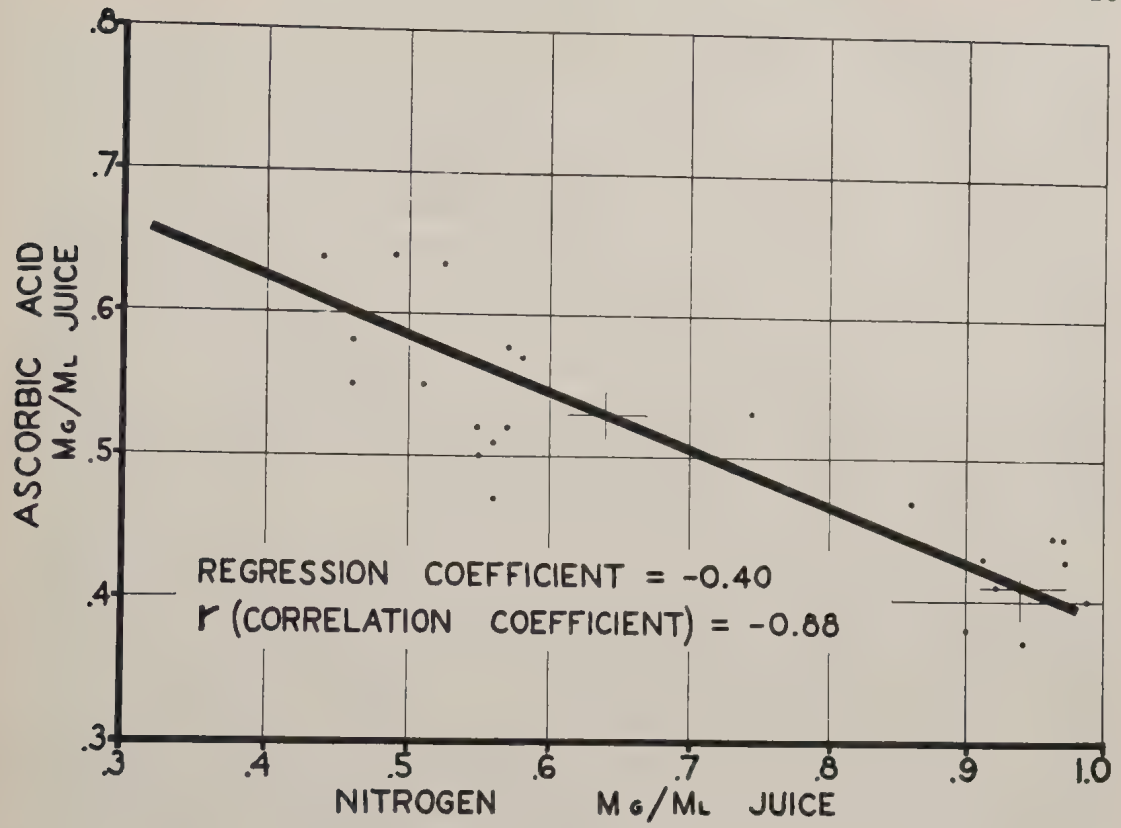


Fig. 26. Regression of vitamin C on nitrogen in grapefruit juice. (From Jones *et al.*, 1945.)

tree in a grove varied greatly in content. However, Ross (1941) found variations in vitamin C content in fruit from trees in different areas, as well as from trees in the same grove. The amount of vitamin C was correlated with differences in the acidity of the juice; the higher the acidity, the higher the vitamin C content.

Harding and Thomas (1942) reported that Foster Pink had a significantly higher vitamin C content than Marsh when both were on sour-orange rootstock. In general, however, Harding and Fisher (1945) reported that the rootstock on which grapefruit was grown had a slight effect on the concentration of ascorbic acid found in the fruit. When the ascorbic acid values were reported as milligrams per milliliter of juice, the amounts were slightly greater when the Marsh and Duncan varieties were on sour-orange than when they were on rough-lemon rootstocks. In ripe grapefruit, small differences were found in the concentrations of ascorbic acid in Marsh fruit on sour orange and in Duncan fruit on rough lemon and sour orange.

Jones *et al.* (1945) found that fruit grown and matured under conditions of declining nitrogen content had more vitamin C in the juice than fruit from high-nitrogen plots. This relationship is graphically illustrated in figure 26, in which the vitamin C content is plotted against the nitrogen in the juice.

The coefficient of correlation between these 2 variables is –0.88, showing a highly significant inverse relation. They reported that sunlight plus nitrogen made favorable conditions for the production of sugars which were convertible into vitamin C.

Hamner (1945) concluded that the vitamin C content of plants was so tied up with such factors as varietal differences, stage of maturity, and climatic conditions that soil conditions and fertilizing practices would have little detectable influence.

Aberg and Ekdahl (1948) stated that plants "right" in nitrogen might decrease the amount of carbohydrates available for the manufacture of vitamin C. This was contrary to the statement by Jones *et al.* (1945), cited above. The amount of nitrogen in the tissues and the time of year are probably the controlling factors.

Vitamin C in Stored Fruit. Fresh citrus fruits are universally recommended, and have proved to be popular and valuable sources of vitamin C. The immediate question of importance is: How stable is vitamin C in fresh citrus fruits under storage and transportation conditions? It is important to both producers and consumers to know to what extent vitamin C is lost during the time interval between harvesting and marketing of the fresh fruit. At present, these problems are of particular interest to the author with respect to grapefruit, realizing that the factors that influence the stability of vitamin C in oranges and lemons are also highly applicable to grapefruit.

It is well known that vitamin C is an oxidizing-reducing agent. This means that under certain conditions, vitamin C reacts with oxygen (oxidation) and under other conditions with hydrogen (reduction). In living tissues, vitamin C usually exists in the reduced form. In many edible plants, vitamin C is oxidized with relative ease, and this usually happens when food is excessively exposed to air. Consequently foods that have a high concentration of vitamin C in the oxidized form cannot serve as effective sources of the active vitamin.

French and Abbott (1940), in the study mentioned in the preceding section, found that vitamin C increased during the first month in storage, and then slowly decreased. Sergeev (1946), in Russia, reported that in fruit stored in light, with access to air, vitamin C was rapidly destroyed, but when stored in the dark without air, 93.3 per cent of it was retained after 90 days.

Vitamin C is well stabilized in intact citrus fruits. This statement was originally substantiated in the 18th century by the classic experiments of James Lind, who showed incidentally that the antiscorbutic factor was retained in citrus fruits during lengthy sea voyages.

Evendon and Marsh (1948) noted that the oxidation of ascorbic acid in orange juice in the liquid state was a first-order reaction whose rate was a function of the temperature. They also observed that ascorbic acid was slowly oxidized by atmospheric oxygen at less than room temperatures.

In studying the effect of low temperature on the physiology of grapefruit, Demoise and Miller (1959) observed that no significant changes in the ascorbic acid content of the pulp segments of Marsh and Thompson grapefruit occurred when they were stored for 4 weeks at 10° C. and for 4 and 6 weeks at 5° C. (50 and 41° F.). During the storage period, ascorbic acid in the peel increased in 1 lot of Marsh fruit, decreased in another, and increased in the peel of the Thompson fruit.

The stability of vitamin C in fresh Valencia oranges under various storage conditions at different temperatures was observed by Pritchett (1962). His analysis of the juice from representative samples showed a decrease in ascorbic acid concentration of 0 to 7 per cent, which occurred during storage periods of 4 to 8 weeks under different combinations of refrigeration and room temperature. In 1 experiment where the samples were held for 2 weeks in iced storage, and 2 weeks at room temperatures ranging from 50 to 85° F. (10 to 29.4° C.), the ascorbic acid concentration never decreased more than 4 per cent—equivalent to a loss of 2 milligrams per 100 milliliters of juice under these experimental conditions.

Hamersma (1938) investigated the temperature–time factor in relation to the stability of vitamin C in oranges under storage conditions. He held Valencia and navel oranges in storage at 38° F. (3.3° C.) for 12 weeks, and subsequently analyzed representative samples for vitamin C at 2-week intervals. The data from these experiments showed no decrease in vitamin C (mg per 100 ml of juice) for the entire storage period.

Vitamin C in Processed Products. Numerous studies have been made on the vitamin C content of canned and bottled juice. Roberts (1937) found that the loss of vitamin C from dairy-type beverages (glass bottles with cardboard stoppers) was rapid, even at cold-storage temperatures.

Floyd and Fraps (1942) reported finding an average of 33.7 milligrams of vitamin C per 100 grams in 109 samples of canned grapefruit juice, compared with an average of 41.2 milligrams in 21 samples of freshly reamed juice from tree-ripened fruit. Ross (1944) found that canned grapefruit juice stored at room temperatures for 12 months kept 70 to 80 per cent of its vitamin C.

Moore *et al.* (1944*b*) found that the retention of vitamin C in juice after being stored for 6 months in glass bottles at 80° F. (27° C.) was 75.1 per cent; in cans, 82.9 per cent. Other samples stored for the same length of time at 40° F. (4.4° C.) retained, in bottles, 89 per cent; in cans, 97.1 per cent. They concluded that the storage temperature had a more significant effect on the retention of vitamin C than the length of the storage period (figure 27). In the following year, these authors (Moore *et al.,* 1945) reported the average retention of vitamin C in canned, unsweetened grapefruit juice for 12 central Florida canneries to be 95, 90, and 83 per cent of the original amount after 2, 4, and 6 months' storage. In another published report, Moore *et al.* (1944*a*)

showed conclusively that 97 per cent of the original vitamin C content of grapefruit juice was retained during commercial processing.

In the commercial canning of grapefruit juice, the same group of authors (Wiederhold *et al.,* 1945) determined the individual factors in plant operation that are most essential to the retention of vitamin C in the final product. These contributory factors are: the absence of copper and other metals that are known to catalyze the oxidation of ascorbic acid; to avoid, as much as possible, the absorption of air into juice during any step in the process; to de-aerate the juice thoroughly; to flash-pasteurize with tubular rather than kettle-type equipment; to reduce oxidation of the ascorbic acid, produce high vacuum in cans, and reduce head space to a minimum; and finally, to place the product in storage at 40° F. (4.4° C.).

Wagner *et al.* (1945) determined the vitamin C retention of the juice from 12 canneries in the Rio Grande Valley of Texas during each successive proc-

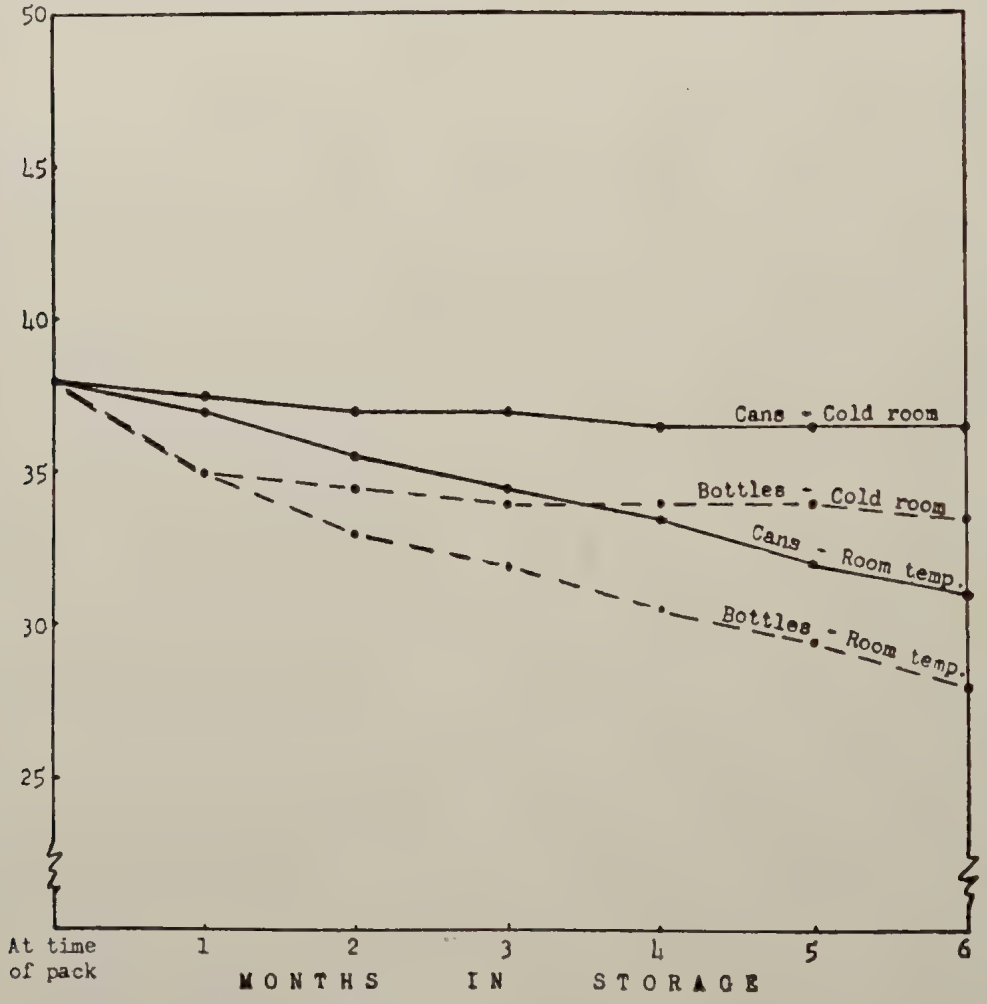


Fig. 27. Decreases in ascorbic acid in unsweetened grapefruit juice packed in tin and glass and stored under cold-room and room-temperature conditions. (From Moore *et al.,* 1944*b*.) *Left margin:* mg per 100 ml.

essing treatment. The retention during the active canning process ranged from 92.2 to 99.6 per cent, with a mean of 96.7 per cent. After a 40-day storage period at an average of 72° F. (22.2° C.), the samples retained a mean of 91.3 per cent of the vitamin C present in the freshly canned juice.

Lamb (1946) tested juice from 3 California and 5 Arizona canneries, and found that during the canning operations, the retention of vitamin C varied from 91.6 to 107.5 per cent of the original, with a mean of 98 per cent.[1] During storage at 70° F. (21° C.) for 18 months, the juice lost its vitamin C at a constant rate of about 1 per cent a month. Tests for iron, reductones, and dehydroascorbic acid in the stored samples indicated that these substances were either absent, or were present in insufficient amounts to interfere with the determination of vitamin C.

Moore (1949) found the following percentages of retention after 18 months of storage: at 76° F. (24.4° C.), 47 per cent in bottles and 58.4 per cent in cans; at 40° F. (4.4° C.), 86 per cent in bottles and 94.2 per cent in cans. At 76° F., the bottled juice showed slight darkening after 6 months and noticeable darkening after 18 months, while the canned juice showed only slight browning after 18 months. At 40° F., the bottled juice showed slight browning after 12 months, while the canned juice retained its normal color after 18 months. Canned juice had better flavor on all dates than bottled juice.

The effects of storage temperatures on the retention of ascorbic acid in grapefruit juice and segments were determined by Sheft *et al.* (1949). They demonstrated that 93 and 94 per cent of ascorbic acid was retained by grapefruit juice and segments at 50° F. (10° C.) during storage periods of 18 and 24 months, respectively. The retention of ascorbic acid decreased with an increase of storage temperature. Their data seem to indicate that a temperature above 65° F. (18° C.) will produce a significant reduction in ascorbic acid (table 54).

Concerning canned segments, Wolfe *et al.* (1949) stated that no significant losses of vitamin C occurred, with or without 40 per cent sugar syrup. The percentage of retention of vitamin C after 1 to 9 months of freezer storage ranged from 93 to 102 [*sic*] per cent in plain-packed fruit and 96 to 102 per cent in the syrup-packed product.

Veldhuis (1951) stated that more than 80 per cent of the original vitamin C content was found in pasteurized canned juice after 6 months' storage at a little above room temperature, provided the juice had not been in contact with either copper or brass. Frozen juice concentrates retained 95 per cent of their vitamin C after 6 months. Stainless steel is now used almost exclusively in processing citrus products.

[1] When percentages in excess of 100 are stated, they represent means or averages of the cited author's experimental determinations. These may be caused by errors of random sampling or by probable errors in the methods used to determine the values. We cite them as originally given.

Enzymatic Relationships. Somogyi (1945*a, b*) stated that the substance that protects vitamin C from destruction (degradation) is the most active of such substances found in plants. Its action is independent of whether vitamin C is being degraded by ascorbic oxidase, phenol polyphenolase, peroxidase, or copper.

Hussein (1944) found that ascorbic acid did not affect the oxygen uptake of orange tissues at pH 5, and that the ascorbic acid added to the reaction medium was fully recovered. Ascorbic acid oxidase, the enzyme that could be expected to react with ascorbic acid, has never been found in orange juice.

Steinman and Dawson (1942) concluded that hydrogen peroxide was not a reaction product of vitamin C when catalyzed by the copper-protein vitamin C oxidase.

Vitamin C in the Pummelo. Su and Liu (1938) reported that the Chinese pummelo (commonly called shaddock) was rich in vitamin C.

Hou (1936) tested 35 varieties of Chinese citrus, and found that the pummelo contained the highest percentage of vitamin C.

Other Vitamins

As a source of vitamins, grapefruit is chiefly valued for its ascorbic acid content, but it also contains several others, as noted in the following text.

Vitamin A. It should be borne in mind that wherever the term "vitamin A" is used in the text or tables, the vitamin came from plant tissues, and should, therefore, be referred to as "provitamin A" or as "carotene." Most of the common fruits contain some carotene. Carotenes and other carotenoids as pulp pigments are discussed starting on page 183 of this chapter.

TABLE 54

Ascorbic Acid Retention of Canned Grapefruit Juice and Segments Stored 18 and 24 Months at 3 Temperatures.*

Months	Temperatures		Ascorbic acid retained in canned grapefruit (per cent)	
	Degrees F.	Degrees C.	Juice†	Segments‡
18	50	10	93	94
	65	18	84	86
	80	27	62	57
24	50	10	94	87
	65	18	82	77
	80	27	57	46

*From Sheft *et al.* (1949).

†Each figure represents the average from analyses of 3 packs; each analysis was done on an aliquot of a 6-can sample.

‡Six cans from 1 pack were mixed for analysis.

Morgan and Chaney (1924) reported that grapefruit juice, fresh or concentrated, contained very small amounts of vitamin A when tested on young rats that had been reduced to a state of vitamin A deficiency.[2] Willimott and Wokes (1927) and Eddy *et al.* (1932) found no vitamin A in grapefruit. Fraps and Treichler (1933) estimated 0.1 unit per gram, 2.8 units per ounce, or 4.5 units per pound.

Thiamin (Vitamin B_1). The importance of thiamin for organisms appears to rest in the fact that it is very necessary for bringing about the decomposition of carbohydrates. A deficiency of this vitamin not only prevents normal growth, but may result in loss of appetite and the occurrence of several ailments, such as neuritis and beri-beri.

Osborne and Mendel (1920) were among the early discoverers of thiamin in citrus fruits, working with oranges and grapefruit. They used the biological assay method, and concluded that the amount of thiamin in these fruits was similar to that usually found in cow's milk. Willimott and Wokes (1927) tested grapefruit flavedo and juice, and found thiamin. Eddy *et al.* (1932) reported that they found thiamin in moderate amounts (14 Sherman units per 100 grams) in canned grapefruit. Roehm (1935) stated that the raw pulp of grapefruit grown on the University farm in Arizona contained 0.16 unit of thiamin per gram of tissue. Baker and Wright (1935) found 0.4 IU of thiamin per gram of grapefruit pulp. In terms of crystalline thiamin, Booher and Hartzler (1939) reported 72 μg, or 24 IU, of thiamin per 100 grams of pulp, without the segment membranes. Bailey and Thomas (1942), in Florida, found 35 μg of thiamin per 100 ml of juice from seedy grapefruit and 32 μg from seedless varieties. An investigator in Buenos Aires (Anon. 1945) reported finding 91 μg of thiamin in the edible portion of grapefruit.

French *et al.* (1951), in Florida, found 0.015 milligram of thiamin in 100 grams of the edible portion of grapefruit. As a result of their tests, they concluded that abnormal levels of soil nutrients would significantly affect the vitamin level in only a few instances. On the other hand, they stated that the content might be affected by genetic background and environment.

Riboflavin (Vitamin B_2, Vitamin G). Riboflavin (B_2) is very widely distributed in the cells of plant and animal tissues; therefore, it is not surprising that it is found, though in small amounts, in the grapefruit. Roehm (1935) reported that the raw peel of grapefruit grown on the University farm in Arizona contained 2.5 times as much riboflavin as the raw pulp—1.0 and 0.4 unit per gram, respectively. Lanford *et al.* (1941) found 20 μg per 100 ml of juice of seedy grapefruit, and 11 μg per 100 ml of juice of seedless varieties. In grapefruit size 70, they found 27 μg per fruit. French *et al.* (1951) composited the tissues and juice of the edible portion of the fruit, and found the composite to contain 0.008 milligram per 100 grams.

[2] One "rat unit" is about the same as 1 international unit (IU); i.e., 0.001 milligram.

Niacin (Nicotinic Acid). Nicotinic acid occurs in small amounts in all living cells. French *et al.* (1951) found 0.15 milligram of nicotinic acid per 100 grams in the composited edible portions of a large number of seedling grapefruit in Florida. An investigator in Buenos Aires (Anon. 1945) found 0.27 milligram of nicotinic acid per 100 grams of pulp. Veldhuis and Gordon (1947) tested yeasts that had been grown on grapefruit juice, and found "significant quantities" of nicotinic acid.

Pantothenic Acid. This acid, one of the vitamins in the "B" complex, is apparently present in all protoplasm. Pure pantothenic acid has been produced in crystalline form, having fine, orange-yellow needles. It is soluble in water, 12 milligrams per 100 milliliters. The daily requirement for adults is 2 to 3 milligrams. Eddy *et al.* (1932) found what they termed "vitamin G" (pantothenic acid) to be present in moderate amounts in canned grapefruit juice. Roehm (1935) found 0.4 Sherman units per gram in grapefruit pulp. Daniel and Munsell (1937) reported that 40 Sherman units of the acid were found in fresh, whole Marsh grapefruit in Arizona.

Inositol. Inositol occurs in practically all plants and animals. In plants, the greatest amounts are found in the leaves. It has been found, in certain plants, to stimulate growth and to function as a building unit. Nelson and Keenan (1933) extracted 0.28 gram of inositol from 10 liters of canned grapefruit juice.

For the definition, methods of determination, and occurrence, see the discussion of inositol by Weidlein (1951).

Vitamin P. The term "vitamin P" is used here with reservations. No one has proved definitely that the flavonoids (naringin and naringenin) are true vitamins. However, it has been suggested that they, like carotene, could be considered to be provitamins.

Armentano (1938) stated that in some cases, naringin or naringenin from the grapefruit proved to be more effective in the treatment of capillary fragility than hesperidin, which occurs in the orange and lemon. Higby (1943) gave a historical review of the discovery, chemistry, and medicinal uses of the so-called "vitamin P." Regardless of the terms applied to these substances, they are useful therapeutic agents or drugs.

In a comprehensive experiment, Krehl and Cowgill (1950) ascertained the amounts of biotin, folic acid, pyridoxine, and inositol in citrus juices, and determined the effects of processing and storage on the stability of this group of vitamins. Their data on fresh and canned grapefruit juice are reported in table 55. In this study, their field-collected, fresh-frozen samples were considered to be fresh, as substantiated by the fact that samples of juice immediately analyzed, after collection, for the various vitamins had approximately the same values as the composite samples of the same juice which had been frozen at the same time at –10° C. (14° F.).

TABLE 55

Vitamin Content of Fresh and Canned Grapefruit Juice.*

Grapefruit juice (unsweetened)	Fresh† or canned‡	Total solids (Brix) (per cent)	Total acid (citric) (per cent)	Ascorbic acid		Biotin (mg/100 ml)	Folic acid (mg/100 ml)	Pyridoxine (mg/100 ml)	Inositol (mg/100 ml)	Number of field boxes sampled	Types of fruit
				Reduced (mg/100 ml)	Total (mg/100 ml)						
Plant E.....	F	10.2	1.31	34.2	38.8	0.36	1.8	9.6	112	3,600	Duncan
	C	10.2	1.31	32.0	33.8	.55	2.2	8.0	104		
Plant L.....	F	9.5	1.24	38.0	40.8	.75	0.8	8.4	98	2,500	20 per cent Marsh Seedless
	C	10.7	1.33	37.2	39.0	.80	1.9	8.8	112		80 per cent Duncan
Plant B.....	F	9.5	0.99	30.2	36.3	.71	0.8	10.4	96	4,500	50 per cent Marsh Seedless
	C	10.1	1.15	30.1	36.8	.60	1.5	8.8	92		50 per cent Duncan
Plant F.....	F	9.4	0.86	35.1	38.3	.97	0.9	11.4	102	900	50 per cent Marsh Seedless
	C	9.9	0.91	30.2	38.8	.92	1.7	12.2	92		50 per cent Duncan
Plant J.....	F	9.6	1.07	37.4	39.7	.83	0.8	14.2	100	4,500	67 per cent Duncan
	C	9.8	1.14	36.2	38.3	.80	1.5	18.2	104		33 per cent Marsh Seedless
Plant H.....	F	9.9	1.02	37.8	40.0	.4	0.9	18.0	88	1,800	Seeded
	C	10.6	1.11	40.0	39.3	.7	1.7	15.2	101		

*From Krehl and Cowgill (1950).
†F equals "fresh." Samples stored at −10 to −12° C. (14 to 10° F.) for 2 to 4 months before analyses.
‡C equals "canned." Samples stored at 27.2° C. (81° F.) for 6 to 8 months, then at 6° C. (43° F.) for 2 to 4 months before analyses.

Ascorbic acid and inositol are present in grapefruit juice in nutritionally significant quantities, whereas biotin, folic acid, and pyridoxine are present in such small microquantities that they would hardly be nutritionally significant in the usual amounts of juice that would normally be consumed by a human individual.

The data in table 56 on the vitamins of frozen grapefruit juice and orange-grapefruit blend have been extracted from a comprehensive table on the composition of frozen fruits and vegetables published by Burger *et al.* (1956). These authors asserted that the samples were taken from commercial processing lines in approximately 150 freezing plants in all production areas, and that they were taken at predetermined intervals to provide for representative sampling with reference to variables in weather, harvesting, processing, and grades.

These values should serve to approach, with a considerable degree of accuracy, the mean vitamin contents of the commercial products usually purchased by the consumer.

Minerals

THE EDIBLE PORTION of the grapefruit has been found to contain many of the minerals that should be included in a well-balanced human diet.

Traub *et al.* (1929) found that the mineral constituents in Marsh grapefruit juice tested from November 19 to December 19, 1928, averaged, as percentages of fresh weight: total ash, 0.33; insoluble ash, 0.039; lime, 0.014; magnesium, 0.015; phosphoric acid, 0.035; and potash, 0.19.

Jones (1931) reported, in several varieties of grapefruit from different places, an average of 0.42 per cent ash in the juice, including 0.40 per cent in that from Florida and 0.40 per cent in that from California. Table 57 gives the kinds and amounts of minerals found in grapefruit pulp by Jones, and in whole fruit and juice by Sherman (1941).

For the amounts of mineral constituents found by Hardy and Rodríguez (1935) in whole fruit, fruit juice, peel ash, and juice ash of Trinidad grapefruit, see table 58. They tested and compared the ash content of the juice and peel from grapefruit grown on lime land (El Quemado estate) and on acid land (Maqueripe estate). The results given in the table show that both juice and peel of "lime-land" fruit were less rich in nitrogen and potash, but richer in lime and, with juice at least, in phosphate, than were those of "acid-land" fruit. High nitrogen and possibly high potash contents seem to have been associated with relatively low sugar content and low sugar:acid ratio.

Haas and Klotz (1935) found that both on a fresh-weight and on a dry-weight basis, the stem half of grapefruit contained higher percentages of calcium and potassium and less phosphate than the stylar half.

TABLE 56

VITAMIN COMPOSITION OF FROZEN GRAPEFRUIT JUICE AND ORANGE–GRAPEFRUIT BLEND.*
(Average values, followed by maximum and minimum.)

Products	Sets tested	Ascorbic acid			Vitamins (mg/100 grams)						
		Reduced	Dehydro	Totals	β-Carotene	Folic acid	Niacin	Pantothenic acid	Riboflavin	Thiamin	Vitamin B_6
Grapefruit juice.....	8	36.1	2.0	38.1	0.004	0.002	0.26	0.162	0.006	0.048	0.014
		41.5	5.5	43.3	.006	.003	.34	.197	.007	.057	.015
		31.5	0.1	34.6	.003	.001	.21	.120	.004	.040	.012
Orange–grapefruit blend.....	8	41.0	2.1	43.1	.004	.003	.31	.150	.008	.064	.024
		47.0	3.7	48.7	.006	.004	.39	.187	.012	.074	.027
		32.3	0.2	34.2	.003	.002	.23	.118	.006	.054	.020

*Adapted from Burger *et al.* (1956), their table 1.

Roberts and Gaddum (1937) analyzed the ash of juice from Marsh and from a seedy variety of grapefruit, with the results shown in table 59. They also tested for 18 other elements, such as copper, lead, manganese, silver, cobalt, and zinc. Nearly all of these were detected, but only in very small amounts. The data in their table 4 (not reproduced here) show the alkalinity of the ash for Marsh to be 3.06 and for the seedy variety, 5.90. The alkali numbers for these 2 varieties were found to be 14 and 14.20, respectively.

Chace *et al.* (1940) found a minimum of 0.390 per cent and a maximum of 0.460 per cent ash in Cuban (Toronja) grapefruit juice.

Harding and Fisher (1945) determined the principal constituents of the ash of Marsh and Duncan grapefruit from 14 experimental plots in 1941–42. The samples came from Marsh on rough lemon in 4 areas, and on sour orange in the same areas. The Duncan was on the same 2 rootstocks, but samples were taken from only 3 instead of 4 of the above areas. The results of these tests are summarized in table 60. The Duncan pulp consistently contained a higher percentage of ash and of potassium than that of the Marsh. The fruit of both contained a slightly greater amount of potassium when on sour-orange rootstock. Rootstock and variety had little if any effect on the content of calcium and magnesium in the fruit. (For more detailed results of these tests, see Harding and Fisher's tables 13 through 33.)

Figures 28 and 29 (pp. 124–125) show clearly the proportion of the individual mineral elements in the ash of various fruit components: peel, pulp, and juice. Calcium composed nearly 20 per cent of the total ash of the peel, about 8 per cent of the ash of the pulp, and about 2.5 per cent of the ash of the juice. A relatively small quantity of the total calcium was soluble in the juice. Thus, the highest concentration of calcium was present in the peel, followed by that in the pulp, including both juice vesicles and juice.

For a given district, the juice contained a higher percentage of its ash as magnesium than did either the peel or pulp. The magnesium concentration never appeared in greater amounts than 5 per cent of the ash constituents. The potassium ion existed in grapefruit in greater concentrations than

TABLE 57

INORGANIC COMPOSITION OF GRAPEFRUIT.*

Portion of fruit	Minerals (as percentages of fresh weight)							
	Calcium	Magnesium	Potassium	Sodium	Phosphorus	Sulfur	Chloride	Iron
Pulp	0.021	0.009	0.161	0.004	0.020	0.010	0.005	0.0003
Whole fruit	.017	.010	.198	.004	.018	.008	.003	.0003
Juice	.010	.008	.139	.005	.017	.005	.005	.0002

*Data on pulp are from Jones (1931); those on whole fruit and juice are from Sherman (1941), his table 63.

TABLE 58

COMPOSITION OF TRINIDAD GRAPEFRUIT.*

Parts of fruit and constituents	El Quemado estate†		Maqueripe estate	
	Choice	Culls	Choice	Culls
WHOLE FRUIT				
Average weight per fruit (grams)	435	437	384	404
Average density (gm/cc)	0.943	1.043	0.993	0.946
Rind and albedo, per cent	22.9	23.8	20.5	25.6
Juice and pulp, per cent	76.8	75.9	79.2	74.2
Total dry matter, per cent	12.0	14.5	13.8	14.5
Total ash, per cent	0.40	0.54	0.44	0.48
JUICE				
Degrees Brix (total solids)	9.10	8.60	9.23	8.90
Degree of acidity (pH)	3.25	3.26	3.10	3.27
Total ash, per cent	0.18	0.24	0.20	0.22
Total sugars, per cent	7.3	6.5	6.9	6.8
Reducing sugars, per cent	4.9	4.8	5.1	5.0
Citric acid, per cent	1.01	0.96	1.11	1.04
Ratios, Brix:acid	9.0	9.0	8.3	8.5
Ratios, sugar:acid	7.2	6.7	6.2	6.5
RIND ASH				
Ash in dry matter (per cent)	4.1	4.2	3.8	3.7
Nitrogen (N), per cent ash	14.6	18.3	20.1	23.9
Phosphate (P_2O_5), per cent ash	3.8	4.6	3.7	4.6
Potash (K_2O), per cent ash	23.0	30.6	34.2	35.7
Lime (CaO) per cent ash	35.9	39.2	23.4	25.1
Ratios, $K_2O:P_2O_5$	6.0	6.6	9.1	7.7
Ratios, K_2O:CaO	0.6	1.0	1.5	1.4
Ratios, $N:P_2O_5$	3.8	4.0	5.3	5.1
Ratios, $N:K_2O$	0.6	0.6	0.6	0.7
JUICE ASH				
Ash in dry matter (per cent)	2.3	3.1	2.5	2.8
Nitrogen (N), per cent ash	23.4	23.9	25.6	27.1
Phosphate (P_2O_5), per cent ash	15.2	12.9	11.5	12.5
Potash (K_2O), per cent ash	49.2	48.4	53.3	52.7
Lime (CaO), per cent ash	10.1	6.7	5.5	6.1
Ratios, $N:P_2O_5$	1.5	1.8	2.2	2.2
Ratios, $N:K_2O$	0.5	0.5	0.5	0.5
Ratios, $K_2O:P_2O_5$	3.2	3.7	4.7	4.3
Ratios, K_2O:CaO	4.8	7.2	9.9	8.8

*Adapted from Hardy and Rodríguez (1935).
†El Quemado estate was regarded as "lime-land," while the Maqueripe estate was "acid-land."

TABLE 59

PRINCIPAL ASH CONSTITUENTS IN THE JUICE OF GRAPEFRUIT.*

Elements	Percentages in juice		Percentages in ash	
	Marsh grapefruit	Seedy grapefruit	Marsh grapefruit	Seedy grapefruit
Potassium	0.089	0.171	40.9	41.4
Phosphorus	.010	.030	4.82	7.04
Calcium	.008	.012	3.64	2.94
Sulfur	.002	.004	0.744	0.871
Magnesium	.005	.009	2.08	2.18
Sodium	.004	.007	1.86	1.71
Iron	.00009	.00008	.039	.020
Aluminum	.0003	.0007	.139	.172
Carbon	.019	.037	8.74	8.90
Oxygen (calculated)	.093	.11	42.7	45.8
Chloride	.001	.002	.531	.548

*Adapted from Roberts and Gaddum (1937), their table 3.

any other mineral constituent. The juice contained, on the average, more than 40 per cent of its ash as potassium, and the pulp contained slightly less than 40 per cent. The lowest concentrations were recorded in the peel. The sodium ion existed in grapefruit in the lowest concentrations of any cation reported in these experiments. The amounts of sodium in the tissues occurred in the descending order of juice > pulp > peel. The average sodium value for juice from the Riverside area amounted to 2.61 per cent (ash-weight basis), and that for juice from the Brawley area, 1.51 per cent.

In relation to the SO_4 ion, the pulp contained the highest concentration, followed by juice and then by peel. The highest average value (10.33 per cent) occurred in the pulp from the Riverside (California) area, and the lowest concentration (6.04 per cent), in the juice from the Brawley (California) area. The peel contained the least phosphate of the 3 tissues, while the juice and pulp were not significantly different in phosphate content.

Fatty Constituents

FATS AND OILS occur in very small amounts in the pulp and in high proportions in the seeds of grapefruit (see p. 215). There appears to have been comparatively little research work done on the fatty constituents. Because of their peculiar nature, they have been difficult to extract from citrus fruits. Some authors refer to the fatty constituents as "lipids" and others as "crude fats." "Crude fat" is the term usually applied to that group of substances which may be extracted by ether, and consists principally of fats and oils. In some cases, this also includes waxes, chlorophyll, and similar substances. In this section, the terms "lipid" and "fat" follow the authors' usage.

[*Turn to page 126.*]

TABLE 60

SEASONAL CHANGES IN SOME OF THE PRINCIPAL ASH CONSTITUENTS IN THE FLESH OF FLORIDA GRAPEFRUIT, 1941–42.*
(Each value is based on the fresh weight of 30 to 40 fruits.)

Variety, rootstock, and location	Potassium (per cent)			Calcium (per cent)			Magnesium (per cent)			Phosphorus (per cent)			Manganese (ppm)			Iron (ppm)		
	Nov. 20–25, 1941	Feb. 12–17, 1942	May 6–11, 1942	Nov. 20–25, 1941	Feb. 12–17, 1942	May 6–11, 1942	Nov. 20–25, 1941	Feb. 12–17, 1942	May 6–11, 1942	Nov. 20–25, 1941	Feb. 12–17, 1942	May 6–11, 1942	Nov. 20–25, 1941	Feb. 12–17, 1942	May 6–11, 1942	Nov. 20–25, 1941	Feb. 12–17, 1942	May 6–11, 1942
MARSH ON ROUGH LEMON																		
Davenport	0.095	0.099	0.083	0.014	0.008	0.008	0.012	0.010	0.009	0.012	0.012	0.014	0.16	0.12	0.19	2.7	4.0	2.5
Bradenton	.088	.077	.063	.019	.012	.012	.010	.008	.008	.017	.015	.016	.11	.17	.15	3.0	3.2	2.2
Fort Pierce	.084	.084	. . .	.016	.011	.013	.011	.008	.010	.015	.015	.016	.03	.11	.26	6 1	6.7	4.2
Homestead	.096	.110	.076	.018	.013	.014	.010	.007	.009	.013	.014	.015	.13	.11	.15	3.4	5.5	4.7
Averages	.091	.093	.074	.017	.011	.012	.011	.008	.009	.014	.014	.015	.11	.13	.19	3.8	4.9	3.4
MARSH ON SOUR ORANGE																		
Davenport	.126	.125	.119	.015	.010	.011	.011	.009	.010	.013	.012	.012	.14	.11	.15	2.5	3.5	2.5
Bradenton	.108	.104	.130	.019	.012	.015	.012	.009	.011	.019	.019	.021	.16	.17	.24	2.4	...	6.2
Vero Beach	.093	.109	.100	.021	.010	.011	.011	.007	.011	.016	.016	.016	.20	.16	.10	5.6	2.3	4.1
Homestead	.095	.094	.080	.018	.014	.013	.009	.008	.007	.013	.013	.013	.13	.15	.16	4.0	3.3	4.4
Averages	.106	.108	.107	.018	.012	.013	.011	.008	.010	.015	.015	.016	.16	.15	.16	3.6	3.0	4.3
DUNCAN ON ROUGH LEMON																		
Davenport	.142	.116	.142	.018	.009	.011	.013	....	.010	.013	.012	.013	.04	.03	.19	2.2	3.0	2.0
Palmetto	.128	.112	.118	.026	.014	.016	.011	.010	.010	.916	.016	.018	.18	.15	.21	4.0	5.2	4.2
Fort Pierce	.113	.123	.120	.017	.010	.011	.018	.009	.010	.017	.017	.016	.17	.10	.10	5.8	3.7	5.7
Averages	.127	.117	.127	.020	.011	.013	.014	.010	.010	.015	.015	.016	.13	.09	.17	4.0	4.0	4.0
DUNCAN ON SOUR ORANGE																		
Davenport	.131	.122	.147	.015	.011	.011	.012	.010	.010	.013	.012	.014	.11	.15	.18	1.4	3.2	2.1
Palmetto	.115	.128	.111	.024	.014	.012	.012	.010	.011	.014	.014	.016	.14	.14	.16	4.3	5.0	5.3
Vero Beach	.178	.128	.127	.022	.014	.014	.013	.008	.011	.018	.019	.017	.19	.21	.24	6.4	7.0	3.8
Averages	0.141	0.126	0.128	0.020	0.013	0.012	0.012	0.009	0.011	0.015	0.015	0.016	0.15	0.17	0.19	4.0	5.1	3.7

*Adapted from Harding and Fisher (1945).

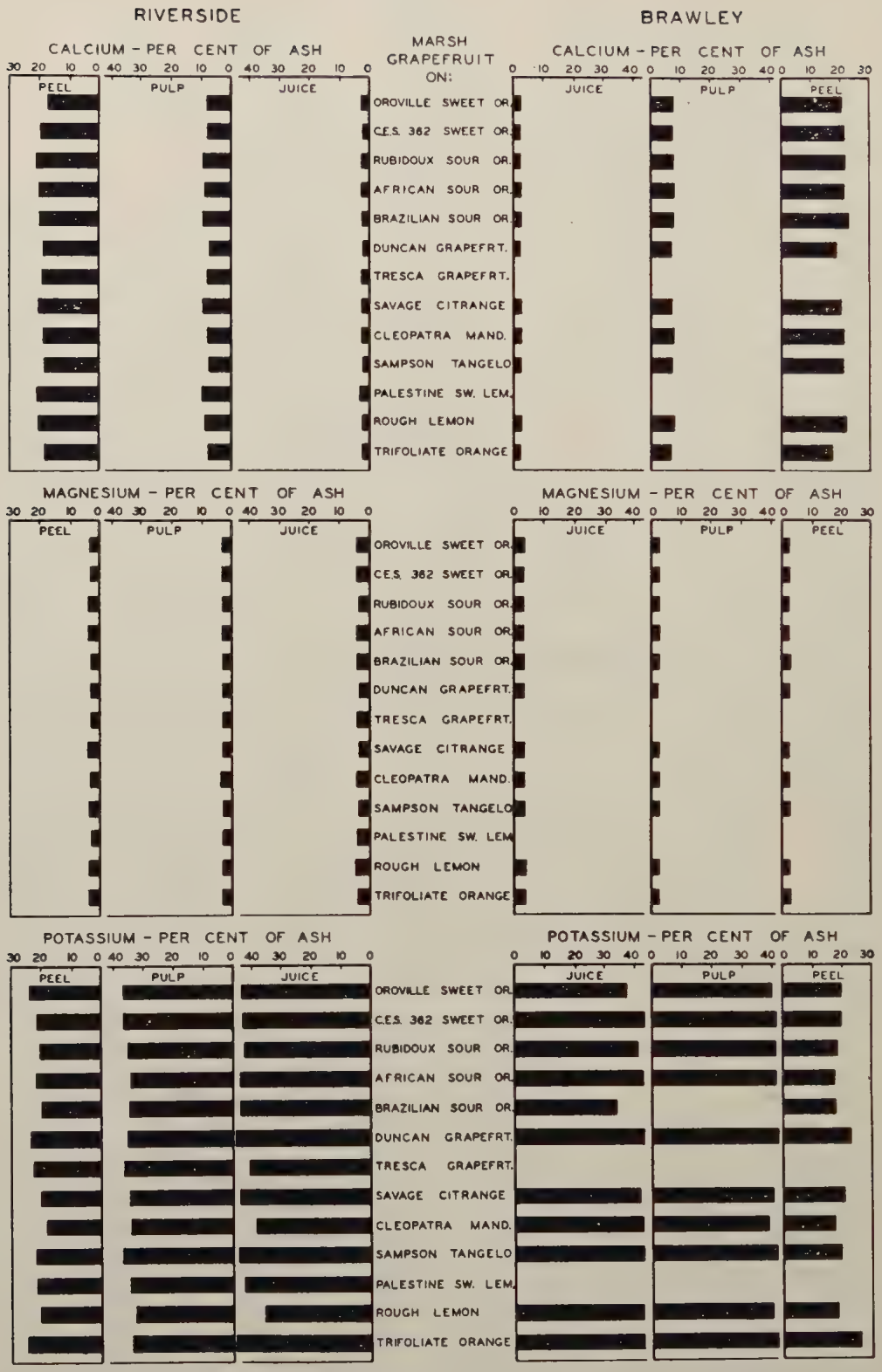


Fig. 28. Relative percentages (ash-weight basis) of calcium, magnesium, and potassium in the peel, pulp, and juice of mature grapefruit from different rootstocks in plots at Riverside and Brawley, California. Fruit was picked at Riverside May 19, 1938, and at Brawley on November 8. (Continued in figure 29.) (From Sinclair and Bartholomew, 1944.)

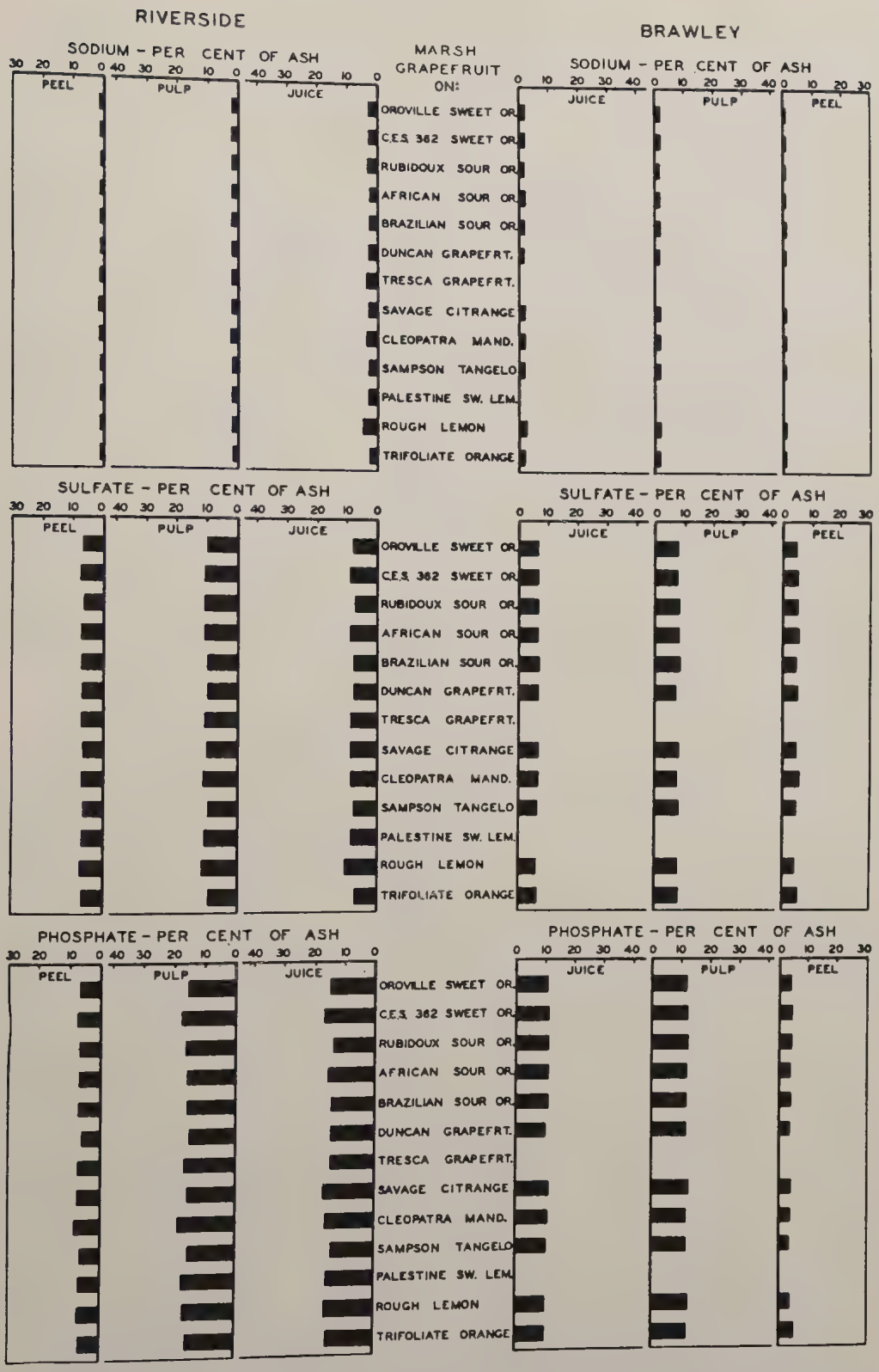


Fig. 29. Relative percentages (ash-weight basis) of sodium, sulfate, and phosphate in the peel, pulp, and juice of mature grapefruit from different rootstocks in plots at Riverside and Brawley, California. Fruit was picked at Riverside May 19, 1938, and at Brawley on November 8. (Continued from figure 28.) From Sinclair and Bartholomew, 1944.)

Lipids are probably more widely distributed throughout the tissues of the plant than any other fatty substance. They are thought by various investigators to play important roles in absorption, secretion, respiration, and transpiration.

Swift (1946) devised a method for the extraction of crude lipids that appears to give reliable results. He found that the crude lipid extract of citrus fruits consisted of approximately 66 per cent saponifiable fatty acid esters and 34 per cent unsaponifiable matter. In grapefruit, he found 0.327 to 0.328 gram of crude lipids per 500 milliliters of juice. Comparative tests showed that the reamed juice contained at least 25 per cent more crude lipids than the press juice.

Jones (1931) found 0.2 per cent fat as the average amount in the juice of several varieties of grapefruit.

Davis (1932) suggested that the oils and waxes in the gland in the center of each juice vesicle might affect the flavor and keeping quality of grapefruit and other citrus juices.

Nolte and von Loesecke (1940*a*) and Nolte *et al.* (1942*b*) suggested that the rancidity of canned juice was caused by the successive oxidation of terpenes, fats, and resins, probably in the order named.

Proteins and Amino Acids

CUBAN GRAPEFRUIT (Toronja) was picked in Cuba over half a century ago and sent to Washington, D.C., where its juice was analyzed by Chace *et al.* (1904). The juice contained 0.56 to 0.58 per cent protein.

Cameron *et al.* (1936) stated that if grapefruit were permitted to remain on the tree as long as they would "hang," the fruits would contain from 20 to 25 per cent more nitrogen than if they were picked as soon as they reached horticultural maturity. (This applied also to oranges and lemons.)

Watt and Merrill (1963) gave values for protein in different parts of the edible portions of the grapefruit, some of which were processed, while others were fresh. Their values were collected over a period of years from published and unpublished data, but no author references were given. The data presented in table 61 are taken from their tables 1 and 2. The following values, expressed in household units, are taken from Watt and Merrill's table 3. The results are stated as grams of protein.

Fresh grapefruit juice, 1 cup (246 grams)	1.2
Canned grapefruit juice:	
Unsweetened, 1 cup (246 grams)	1.2
Sweetened, 1 cup (251 grams)	1.3
Grapefruit juice concentrate, frozen:	
1 cup (6 fluid ounces, 202 grams)	3.8

Chatfield and McLaughlin (1928) gave similar tabulated data for proteins in different parts of the grapefruit.

Alcohol-Soluble Nitrogen and Free Amino Acids. Hiwatari (1927) isolated and identified from the pummelo or shaddock (*Citrus grandis*) the 3 nitrogenous compounds, stachydrine, glycocell-betaine (glycine-betaine), and putrescine.

In studying the nitrogenous contents of orange juice, Nelson *et al.* (1933) identified stachydrine, arginine, choline, asparagine, aspartic acid, glutamine, and small amounts of histidine.

Jansen and Jang (1952) isolated cysteine and glutathione, as their s-benzyl derivatives, from orange juice. They found that the major portion of the sulfhydryl compounds of both Valencia and navel orange juices existed as cysteine and glutathione. The determination of cysteine before and after hydrolysis demonstrated that the total sulfhydryl of the concentrates studied was cysteine and glutathione. These results were extended by Miller and Rockland (1952), who estimated the cysteine and glutathione contents of

TABLE 61

PROTEINS FOUND IN DIFFERENT PORTIONS OF THE GRAPEFRUIT.*

Portions, fresh or processed	Protein (grams per 100 grams)	Protein (grams per pound)†
Raw		
Pulp (pink, red, white)	0.5	1.1
All varieties	.5	1.0
Marsh Seedless (California and Arizona)	.5	1.0
All varieties (Florida)	.5	1.1
All varieties (Texas)	.5	1.1
Canned segments in sirup, solids and liquid	.6	2.7
Canned in water pack	.6	2.7
Juice		
Fresh (all varieties)	.5	2.3
Canned		
Unsweetened	.5	2.3
Sweetened	.5	2.3
Frozen concentrated		
Undiluted (unsweetened)	1.9	8.6
Diluted with 3 parts water	.5	...
Grapefruit–orange blends		
Canned		
Unsweetened	.6	2.7
Sweetened	.5	2.3
Concentrate, frozen (undiluted)	2.2	9.5
Concentrate, diluted with 3 parts water	.6	...

*Adapted from Watt and Merrill (1963).
†One pound equals 0.454 kilogram.

fresh and heat-treated grapefruit and other citrus juices (table 62). The fresh juices that were heated showed smaller amounts of both compounds.

Townsley *et al.* (1953) determined the total nitrogen extractable with 70 to 80 per cent alcohol from various tissues of mature grapefruit and other citrus types (table 63). These authors maintained that the free amino acids in the alcoholic extracts probably included the easily hydrolyzable amino acids from the more complex units. The tissues were immediately placed in alcohol after separation from the fruit and stored at zero centigrade (32° F.), which resulted in a great reduction in the period of enzymic hydrolysis.

By the use of small-scale filter-paper chromatography, Underwood and Rockland (1953) determined the relative amounts of nitrogenous constitu-

TABLE 62

CYSTEINE AND GLUTATHIONE CONTENT OF FRESH AND HEAT-TREATED CITRUS JUICE.*

Juice sources	Cysteine			Glutathione		
	Fresh (mg per cent)	Heated† (mg per cent)	Loss (per cent)	Fresh (mg per cent)	Heated† (mg per cent)	Loss (per cent)
Orange (Valencia)	0.55	0.41	25	7.8	5.6	28
Grapefruit	.57	.27	53	5.9	5.7	3
Lemon	.32	.28	13	2.8	2.1	25
Lime	.82	...	..	5.1	...	..

*From Miller and Rockland (1952). Calculated from analysis of sulfhydryl concentrates. These values represent the mean of 4 determinations. The mean deviation from the mean averaged 9 and 12 per cent for glutathione and cysteine determinations, respectively.

†Heated in sealed, enamel-lined tin cans for 15 minutes at 100° C. (212° F.). A commercial pasteurized sample of navel orange juice contained 0.49 mg percentage of cysteine and 2.6 mg percentage of glutathione.

TABLE 63

TOTAL ALCOHOL-EXTRACTABLE NITROGEN.*

(Milligrams per 100 milliliters of extract.)

Portions of fruit	Late Valencia oranges	Early green Washington Navel oranges	Late grapefruit	Tree-matured Eureka lemons
Flavedo	14.48	14.08	15.87	8.85
Albedo	10.91	5.79	7.66	6.86
Carpellary membrane	7.20	6.23	2.22	2.63
Vesicles	22.36	12.86	15.16	9.01
Vascular tissue	14.28	....	9.13	11.41
Seeds	32.93	....	22.62	9.13
Reamed juice	19.36	11.63	13.65	8.33
Centrifuged juice	19.69	11.82	....	....
Chromatophores	23.29	14.05	....	....
Navel	....	14.92	....	....

*From Townsley *et al.* (1953).

TABLE 64

Some Nitrogenous Constituents in Fresh and Processed Citrus-fruit Juice.*

Constituents and related data	Orange					Tangerine		Grapefruit					Lemon			Lime	Tangor	Mandarin			Lemon-orange: sweet orange × tetraploid Lisbon lemon
	Parson Brown	Valencia		Washington Navel		California	Commercial	Marsh (Arizona)	Marsh (California)	Pink (Texas)		Marsh (Florida)	Eureka	Commercial		Lime	Dweet	Kara	Kinnow	Honey	
Sample	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
Condition†	*F*	*F*	*C*	*F*	*C*	*F*	C	*F*	*F*	*F*	*C*	*C*	*F*	*C*	*C*	*F*	*F*	*F*	*F*	*F*	*F*
Constituents‡																					
Aspartic acid	3	3	4	4	3	4	3	4	3	4	4	4	3	4	3	2	3	3	2	2	3
Asparagine	2	2	3	3	3	3	1	3	2	3	2	3	1	3	1	1	3	2	2	1	1
Serine	2	3	4	3	3	3	2	3	3	2	3	3	3	3	3	3	2	3	3	3	3
Alanine	2	2	2	2	2	2	1	3	2	3	3	3	2	2	2	3	2	2	2	2	2
Glutamic acid	1	2	1	2	1	2	1	2	2	2	2	2	2	2	2	3	3	2	2	2	3
Proline	2	4	2	1	2	1	0	2	1	1	1	1	1	1	1	2	4	3	3	3	3
γ-Aminobutyric acid	2	3	2	1	1	2	0	2	2	2	3	2	1	1	1	1	3	2	0	0	2
Glutamine	1	1	0	1	0	1	0	1	1	1	tr	0	0	0	0	0	1	1	1	0	0
Arginine	2	4	4	2	3	3	1	3	2	3	3	3	0	0	0	0	3	3	3	3	2
Histidine	..	tr	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
pH	4.2	3.7	3.5	3.4	3.5	3.6	3.4	3.0	2.8	3.4	3.2	3.0	2.1	2.0	..	2.2	3.3	3.1	3.1	3.6	2.4
Acid	0.39	1.11	1.2	1.12	0.99	0.74	0.76	1.7	2.15	0.93	1.31	1.63	6.58	5.47	..	7.88	1.12	1.56	1.51	0.99	5.86
Brix	12.8	12.2	13.0	12.9	14.7	13.0	13.7	10.6	13.0	9.9	12.7	12.2	9.7	8.5	..	12.2	12.2	18.3	16.6	15.9	11.9
Brix:acid ratios	32.8	11.0	10.8	11.5	14.9	17.6	18.0	6.2	6.1	10.6	9.7	7.5	1.5	1.6	..	1.5	10.9	11.7	11.0	16.1	1.9

*Adapted from Underwood and Rockland (1953).

†*F* and *C* refer to fresh and canned pasteurized juices, respectively.

‡The numbers opposite constituents indicate the relative amounts of constituents estimated by visual comparison of spot intensities on 2-dimensional filter-paper chromatograms. The number "0" indicates that a constituent could not be detected, and that less than approximately 5 mg per cent of the constituent was present.

TABLE 65

DISTRIBUTION OF AMINO ACIDS IN LATE GRAPEFRUIT TISSUES.*
(Arranged in the order of approximate intensity of coloration with the reagent ninhydrin.)†

Flavedo	Albedo	Vesicles	Seeds	Carpellary membrane	Vascular tissue
An	Ac	Ac	An, Gc	Ac, Gc, Ab	An
Gn, Sn, Ac, Gc, Al	An	An	Sn	Gn, Sn, Al	Gn, Sn, Ac, Gc
Ab	Sn, Ab, Gn	Sn, Ab	Ac, Al, Ab	U, Pr	Al, Ab
Pr, U, Vn‡	Gc, Al, Pr	Gn, Ar, U, Gc, Al	Tn, Vn‡	....	U
U, Tn, Pn§	....	Ls	Gn	Vn, Ar, U, Tn‡	....
....	Pn§	Vn, Tn, Pr‡	Pr	Pn§	Vn, Tn, Pr,‡ Pn§
....	Vn‡	Pn§	Ln, Pn	....	....
....	....	....	Ar	....	....
....	....	....	Ty	....	....

*From Townsley *et al.* (1953).
†The amino acids identified were: Al, alanine; Ar, arginine; An, asparagine; Ac, aspartic acid; Ab, γ-aminobutyric acid; Gn, glutamine; Gc, glutamic acid; Ln, leucine; Ls, lysine; Mn, methionine; On, ornithine; Pn, phenylalanine; Pr, proline; Sn, serine; Tn, threonine; Ty, tyrosine; Vn, valine. U indicates several unknowns.
‡Valine and possibly methionine.
§Phenylalanine and possibly leucine groups.

ents in the fresh and processed juices of many commercial varieties of citrus fruits (table 64). The constituents of the fresh and processed juices were significantly different.

Townsley *et al.* (1953) identified and showed the distribution of free amino acids in various grapefruit tissues (table 65). The tests were made by means of paper-partition chromatographic analyses of alcoholic extracts of the fruit tissues. The data indicate that different tissues contained quantitatively different amounts of free amino acids.

Wedding and Horspool[3] determined the free amino acids in the juice of grapefruit grown in the Imperial Valley of California. Samples were taken January 6, 1954. These amino acids were extracted from the centrifuged juice with 80 per cent alcohol. To rid the extracts of alcohol, they were evaporated to near-dryness in a current of air at laboratory temperature. The residue was dissolved in water and diluted to known volume, and aliquots were taken for analysis by filter-paper chromatography. The mean total nitrogen found in 5 samples of grapefruit juice was 0.875 milligrams per milliliter. The total amino nitrogen, calculated from the percentage of nitrogen in the amino acids determined in the juice, amounted to 0.835 milligrams per milliliter; thus, the amino acids accounted for almost all the nitrogen found in the unhydrolyzed centrifuged juice.

[3] R. T. Wedding and R. P. Horspool. Unpublished data.

The components the authors identified and the amounts in milligrams per milliliter of juice were as follows: aspartic acid, 1.042; glutamic acid, 0.233; alanine, 0.204; asparagine, 1.472; serine, 0.270; lysine, 0.711; proline, 0.500; glutamine, 0.193; γ-aminobutyric acid, 0.271; and ornithine, 0.312.

Clements and Leland (1962) made an interesting study on the seasonal changes of the free amino acids in Valencia orange juice which could have an important bearing on the relationship of total nitrogen to the various amino acids in grapefruit juice. Their data in table 66 show the changes in amino acid concentrations and nitrogen values in Valencia orange juice during a 14-month sampling period. The amount of nitrogen equivalent to the amino acids was calculated and compared with the total nitrogen (Kjeldahl nitrogen content).

The amino acid nitrogen varied from 61 to 83 per cent of the total nitrogen during the season. Consequently, the amino acid nitrogen and the Kjeldahl nitrogen increased correspondingly throughout the season. Arginine, γ-aminobutyric acid, and proline were the predominant amino acids in late-season fruit. In fact these acids showed a steady increase in concentration throughout the season.

The total nitrogen content of citrus juices, as determined by the Kjeldahl procedures, includes all forms of nitrogen in the juice. The chief nitrogen constituents are the proteins which are alcohol-insoluble, amino acids, amines, and amides; but the amino acids which are alcohol-soluble compose the largest proportion of the total nitrogen.

Ting (1967) reported that the mean monthly nitrogen content of orange juice increased from 0.065 to 0.120 gram per 100 milliliters in samples selected in 3 Florida processing plants, October through May, 1964–65 (figure 30). The rate of increase in total nitrogen was nearly linear from October to January, but from January through March, it was considerably reduced. After March, the nitrogen increased sharply. These trends were related not only to the length of time the fruit remained on the trees, but also to the increase in fruit maturity. It thus follows that late-maturing fruit had a higher nitrogen content because it remained on the trees longer than the early and midseason varieties. The period from October through January was the time when the early and midseason varieties were maturing, and it was also marked by a rapid increase in the total nitrogen content of the juice. The accelerated rate of increase in nitrogen content between March and May also coincided with the time of maturity for late-season varieties, such as Valencia. The fact that the nitrogen content changed slightly between January and March indicates that the fruit entering the processing plants had a higher proportion of less mature late-season fruit (Valencia). The spring flush of new growth and bloom which occurred during February and March also drew heavily on the nitrogen content of the trees.

TABLE 66

SEASONAL VARIATIONS IN THE FREE AMINO ACIDS IN VALENCIA ORANGE JUICE.*

Constituents	Micromoles per milliliter of juice													
	Sept. 30, 1959	Nov. 2, 1959	Dec. 1, 1959	Jan. 15, 1960	Feb. 24, 1960	Mar. 16, 1960	Apr. 4, 1960	May 4, 1960	June 1, 1960	July 5, 1960	Aug. 29, 1960	Oct. 5, 1960	Nov. 4, 1960	Dec. 12, 1960
Alanine	0.7	0.6	0.7	1.1	1.1	1.1	1.4	1.3	1.7	1.7	2.2	3.0	4.0	4.5
γ-Aminobutyric acid	0.8	0.9	0.9	1.3	2.0	2.1	3.3	2.4	3.3	4.1	5.6	6.8	9.4	10.0
Ammonia	6.0	3.5	2.6	1.5	1.1	1.0	1.1	0.7	0.8	0.6	0.3	0.5	0.3	0.3
Arginine	0.3	0.6	1.4	1.9	2.2	2.6	3.0	4.2	4.4	5.2	6.0	6.9	9.3	12.0
Asparagine	5.2	3.5	2.8	2.6	3.1	2.5	3.6	2.7	2.3	2.8	3.7	3.4	3.9	4.5
Aspartic acid	2.7	3.5	3.1	2.4	2.6	2.3	2.5	2.3	2.5	3.0	3.5	3.6	4.6	5.3
Glutamic acid	1.3	0.9	1.2	0.9	1.2	0.9	1.1	1.3	1.2	1.2	1.0	0.9	0.9	0.8
Glycine	0.1	0.1	0.1	0.2	0.2	0.2	0.2	0.3	0.3	0.4	0.4	0.4	0.7	0.8
Lysine	0.1	0.1	0.1	0.2	0.2	0.2	0.3	0.3	0.4	0.4	0.5	0.6	0.9	0.8
Phenylalanine + tyrosine	0.2	0.2	0.2	0.3	0.2	0.3	0.4	0.5	0.5	0.5	0.5	0.7	0.8	0.8
Proline	1.5	3.5	5.1	9.1	14.2	14.0	19.0	15.0	16.0	17.7	18.5	23.9	34.8	38.2
Serine	3.5	2.0	1.7	1.7	1.8	1.7	2.1	1.8	1.7	1.8	1.9	2.0	2.6	3.2
Unknown VIII†	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.3	0.3
Unknown III†	tr	tr	tr	0.1	0.1	0.1	0.1	0.2	0.2	0.3	0.5	0.8	1.3	1.1
Total micromoles amino acids per ml	22.5	19.5	20.0	23.4	30.1	29.1	38.2	33.1	35.4	39.8	44.7	53.7	73.8	82.6
Total micromoles N per ml (from amino acids)	28.7	24.9	27.1	31.9	40.0	39.6	51.1	48.7	51.3	58.6	66.9	78.4	106.5	123.9
Total micromoles N per ml (Kjeldahl)	46.8	36.6	41.1	46.8	56.2	56.6	70.1	64.6	71.8	77.8	93.8	106.8	136.2	149.0
Milligrams N per ml (Kjeldahl)	0.66	0.51	0.58	0.66	0.79	0.79	0.98	0.90	1.01	1.09	1.31	1.50	1.91	2.09
N accounted for (per cent)	61	68	66	68	71	70	73	75	71	75	71	73	78	83

*From Clements and Leland (1962).
†Expressed as leucine equivalents.

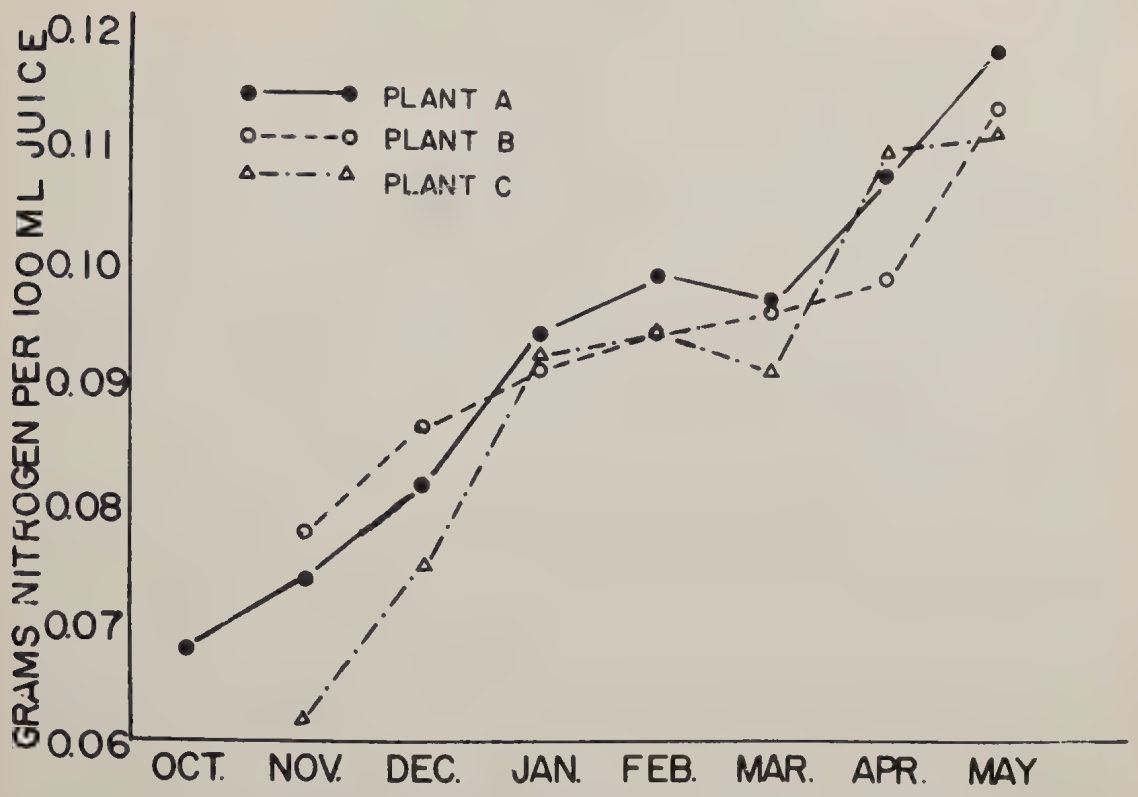


Fig. 30. Seasonal changes in the total nitrogen content of orange juice received at 3 Florida processing plants in the 1964–65 season. Plants *A* and *B* were located in the central ridge area; plant *C*, in the east-coast area. (From Ting, 1967.)

There is a significant correlation between the total soluble solids and the total nitrogen content of orange juice during fruit maturity (Ting, 1967). This relationship undoubtedly holds for grapefruit. Since these 2 variables are chemical components of the fruit that increase significantly during growth and maturation, it is not surprising that the total soluble-solids content is highly correlated with the nitrogen content. For the entire sampling period, October through May, the correlation coefficient was + 0.7, which was highly significant at the 1 per cent level. The relationship between the 2 variables in October was actually a significant negative correlation, which may indicate that other factors, such as rootstock, soil characteristics, and cultural practices, which increase the nitrogen content of the juice, may produce an opposite effect on the soluble solids.

Naringin

In addition to the morphological differences that distinguish it from most other species of citrus, the grapefruit has also an important chemical difference, shared by the pummelo or shaddock: it contains a glycoside called "naringin." Naringin ($C_{27}H_{32}O_{14} \cdot 2H_2O$) is very bitter. It is related to the almost tasteless hesperidin in the sweet orange and lemon, but differs from

both. Its structural formula is shown in figure 31. Compounds of this structure usually occur as glycosides, and they are most abundant in the mesocarp or albedo. They are often classed as plant pigments, even though those found in citrus fruits are usually colorless. These compounds may all be considered as derivatives of γ-pyrone, a ring of 5 carbons and 1 oxygen, with a quinoid oxygen on the carbon para to the oxygen of the ring. The simplest aromatic derivative is benzopyrone, with a plain benzene ring having 1 side in common with the γ-pyrone ring. A phenyl group attached to the carbon adjacent to the oxygen in the pyrone ring gives the real basic unit for the flavone derivatives, 2-phenyl-benzopyrone. The flavonoids are thus derivatives of a C_6—C_3—C_6 skeleton.

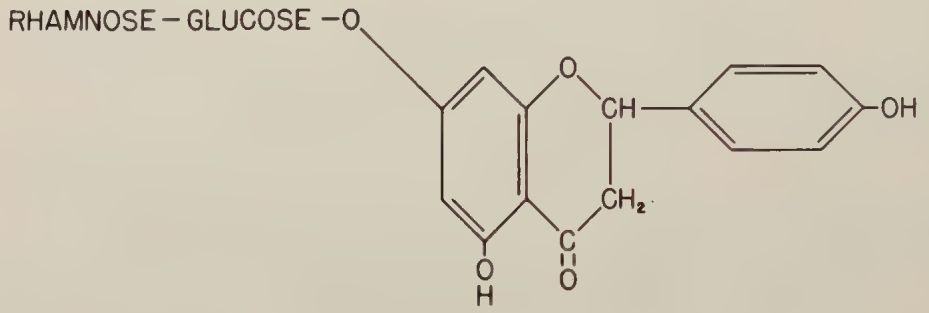


Fig. 31. The structural formula of naringin.

Sugars, usually both rhamnose and glucose, are attached to carbon 7 for citrus flavonoids, with only a few cases where rhamnose alone is attached to carbon 7. When sugars are attached to the flavonoid nucleus, the compounds are called glycosides; when the sugars have been removed or are not present, the term "aglycone" is applied.

The substance now called naringin was first discovered by De Vry in all tissues, mainly in the flowers, of *Citrus decumana* in Java more than a century ago (1857), but he apparently did not publish his findings at that time. Both he and Hoffman (1876*a*, *b*) and later Will (1885; 1887*a*, *b*) conducted extensive researches on this glycoside. According to Hoffman, the name "naringin" came from the Sanskrit *narangi* (Marathi, *naringi*), which means "orange."

Naringin is soluble in alcohol, acetone, and hot water, but only to the extent of about 1 part in 2,000 in water at 20° C. (68° F.). When dried at 110° C. (230° F.), it melts at 171° C. (340° F.), but when crystallized from water, it has an additional 6 molecules of H_2O, and melts at 83° C. (181° F.). The bitterness of this glycoside is so pronounced that it can be detected when 1 part is dissolved in 50,000 parts of water (Braverman, 1949).

Naringin occurs in low concentrations in the juice of mature grapefruit, but in higher concentrations in segment membranes, core, and peel. This section deals chiefly with its occurrence in whole, fresh fruit, juice, and canned

and frozen products. Changes in naringin content during growth and in storage, and the effect of this glycoside on flavor and quality are also discussed. Its occurrence in the peel tissues is discussed in more detail in chapter 3, where methods of extraction, color reactions, and hydrolysis products are also reported. The production of naringin for flavoring and possible pharmaceutical purposes is briefly treated in chapter 6, under "Naringin (Flavonoid)."

Willimott and Wokes (1926, 1927) stated that there was a close relationship between acidity and the sugar and glycoside contents of the grapefruit while maturing.

Fellers (1929) emphasized that naringin crystals sometimes separated out in canned fruit or juice, especially when slightly immature fruit was used. He suggested that in order to keep these bitter-tasting crystals from remaining in the canned segments and juice, the fruit should be carefully prepared, to avoid getting the naringin from the albedo and also from the segment membranes. He added that the more mature the fruit, the less would be the danger of extracting naringin during processing.

Camp *et al.* (1932), who were among the earlier workers to study the causes of unsatisfactory canned citrus products, noted that the bitter taste in the stored products on aging was of glycosidal origin, localized primarily in the albedo, vascular bundles, and segment walls. They found that the intensity of the bitterness was a function of fruit maturity, method of preparation, and after-treatment of the product, and that the glycosides had to be controlled or removed before a satisfactory frozen or cold-stored product could be obtained.

Poore (1934) found 0.06 per cent naringin in grapefruit juice.

Davis (1947) described a colorimetric method for the determination of flavanones in citrus. By this method, he found 0.041 gram of flavanones as naringin per milliliter of fresh grapefruit juice, of which 0.038 gram occurred in the centrifuged juice and 0.003 gram in the sediment. In other portions of the fruit, he found the following amounts, in grams per hectogram: juice vesicles, 0.140; segment membrane, 1.250; albedo, 2.100; flavedo, 0.600; and core, 2.600.

The Davis color method for the determination of flavanones has been used, with some difficulty, to follow the progress of debittering grapefruit juice and other citrus products. Under practical conditions, it has been demonstrated that partial debittering of grapefruit juice occurs before any appreciable disappearance of naringin, as determined by the Davis procedure (Griffiths and Lime, 1959; Thomas *et al.*, 1958). This situation has led to the suggestion that the bitter naringin and the less bitter prunin would give about the same Davis color values without a correspondingly high bitter flavor. The nonbitter naringenin, the final stage of hydrolysis, develops fewer Davis color values than either of the above compounds.

Horowitz and Gentili (1959) reported on the proper use and limitations of the Davis method. They found that under the specified conditions of alkalinity, both naringin and prunin had significantly strong absorption at 427 mμ, while the nonbitter naringenin showed no significant absorption in that region (figure 32). Naringenin could hardly be determined by absorption measurements in the region of 400–500 mμ, and especially if other compounds were present in citrus which might possess strong absorption in this region in alkaline solution. Hesperidin, like naringenin, had only a slight absorption in the wave lengths greater than 400 mμ. Therefore, the Davis method was not applicable to the quantitative determination of hesperidin and naringenin in citrus juices and products between wave lengths of 400–500 mμ.

These authors noted that the absorption curves of naringin and prunin were distinctly different from those of other flavanones, in that they were easily converted to their chalcones in a weakly alkaline solution. The designated band at 427 mμ was ascribed to the ionized chalcone. The transformation to the chalcone was probably a property of flavanones having both a protected 7-hydroxyl and a free 4-hydroxyl group.

Maurer *et al.* (1950) found less naringin in Texas grapefruit than other investigators found in California grapefruit. The pink and red varieties tended, on the whole, to have less naringin than the light-fleshed Marsh and Duncan (table 67). In all varieties, all the various workers found that the percentage of naringin in the fruit decreased as the season progressed. The

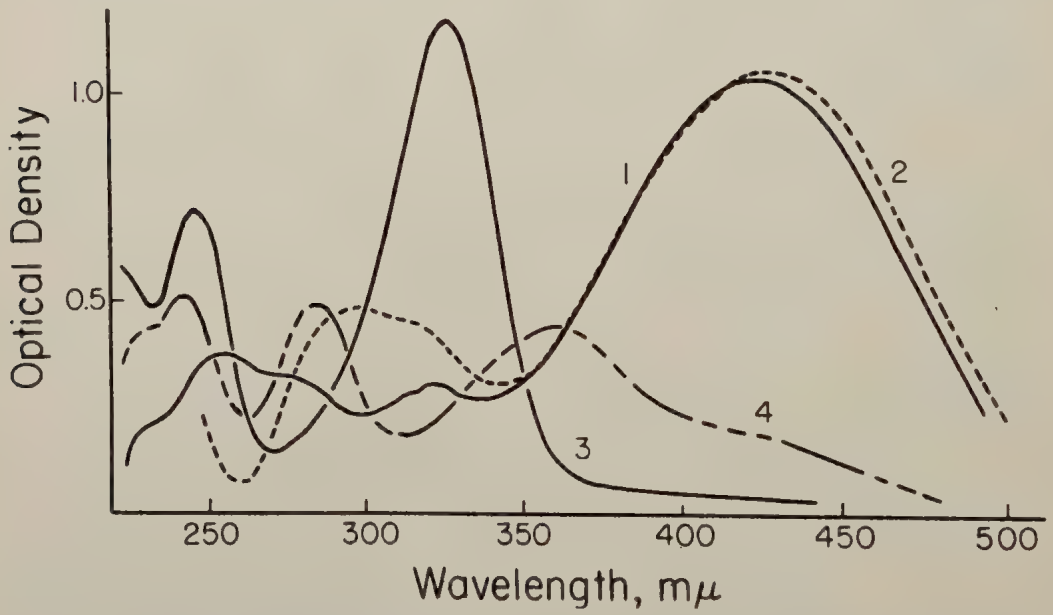


Fig. 32. Absorption spectra in 90 per cent diethylene glycol—0.08 *N* sodium hydroxide. Flavanone concentration: 3.8×10^{-5} M. (1) naringin; (2) prunin; (3) naringenin; (4) hesperidin. (From Horowitz and Gentili, 1959.)

most significant change (decrease) in the percentage of naringin during the harvest season in 1948 was between October 13 and November 17, when the naringin content of the juice dropped, on the average, by 66 per cent. The juice at all times contained less naringin than did the peel tissues, membrane, or core. Maurer and associates stated that when the naringin in the juice samples exceeded 0.070 per cent, the juice was inferior and had a bitter taste, while fruits with juice containing less than 0.050 per cent naringin appeared to have superior flavor and quality.

In most grapefruit-growing countries, the flavor of the juice is considered to be at its best when the naringin percentage is lowest. Other factors, such

TABLE 67

DISTRIBUTION OF NARINGIN IN TEXAS GRAPEFRUIT.*

(All amounts are expressed as percentages except in flavedo and juice.)

Varieties	Dates picked	In flavedo†	In albedo	In section membrane	In core	In juice†
Duncan (white, seedy)	Oct. 13	1.73	4.00	2.92	3.51	0.095
	Nov. 17	0.56	2.52	0.89	1.13	.049
	Dec. 23	0.57	2.00	0.79	1.20	.035
	Jan. 27	0.45	1.49	0.69	1.28	.015
Marsh (white, seedless)	Oct. 13	2.38	5.35	2.91	2.42	.130
	Nov. 17	0.61	2.04	0.74	0.97	.043
	Dec. 23	0.67	2.69	1.35	1.07	.054
	Jan. 27	0.67	2.37	1.38	1.67	.019
Thompson (Thompson Pink)	Oct. 13	5.45	4.90	2.05	1.34	.095
	Nov. 17	0.77	2.43	0.68	0.74	.035
	Feb. 27	0.57	2.11	0.90	1.00	.013
Foster (Foster Pink)	Oct. 13	1.40	4.12	2.10	2.60	.133
	Nov. 17	0.67	2.30	0.93	0.92	.035
	Dec. 23	1.05	3.32	1.41	1.68	.031
	Jan. 27	0.57	2.04	0.84	1.17	.019
Ruby Red (Henninger)	Oct. 13	1.25	4.02	3.07	5.42	.074
	Nov. 17	0.41	2.46	1.15	1.48	.019
	Dec. 23	0.51	4.10	1.16	2.24	.019
	Jan. 27	0.59	2.75	1.27	1.58	.039
Webb (Redblush)	Oct. 13	2.43	4.95	1.70	2.92	.086
	Nov. 17	0.55	2.53	1.26	2.06	.023
	Dec. 23	0.71	3.75	1.56	1.56	.019
	Jan. 27	0.54	1.88	0.99	1.19	.016

*Adapted from Maurer *et al.* (1950).
†In flavedo, naringin is expressed as grams per 100 grams fresh weight; in juice, as grams per 100 milliliters.

as sugars, acids, total soluble solids, pH, esters, oil content, and probably some unknown ones, also affect the flavor. Naringin crystals seldom form in frozen grapefruit as hesperidin crystals do in frozen oranges and lemons.

Kesterson and Hendrickson (1952) found that the naringin content, both in grams per fruit and in distribution among the tissues, became rather constant after the equatorial diameter of the fruit had reached 2 inches (5 cm) (table 68). As the fruit grew larger, the naringin content decreased as a percentage of its over-all weight.

In a later paper, Kesterson and Hendrickson (1953) again reported the relative amounts of naringin in various tissues of grapefruit picked during the growing season, 1951–52 (figure 33). Seasonal variations apparently were not important factors in influencing the distribution of naringin. One exception was noted, in that a slight decrease occurred in the content of the flavedo during the sampling period.

In their 1953 paper, these authors described more clearly the factors that they found to influence the percentage of naringin in grapefruit. Contrary to the results of other investigators, they noted no decrease in the total amount of naringin. Owing to the increase in the size and weight of the fruit, the percentage of naringin decreased as a result of its growth. The percentage in the Duncan variety decreased with fruit growth when calculated either on a basis of dry weight or whole-fruit weight (figure 34).

TABLE 68

MONTHLY VARIATION IN NARINGIN CONTENT OF THE WHOLE FRUIT, AND IN DIFFERENT PARTS OF THE DUNCAN GRAPEFRUIT.*

Dates	Whole fruit			Distribution of naringin (percentages of total)			
	Diameters		Naringin contents (grams)	In juice	In albedo	In flavedo	In rag and pulp
	Inches	Centimeters					
4/28/52.....	0.5	1.2	0.25	...	...	...	...
5/1/51.....	1.2	3.0	1.4	...	...	...	...
6/1/51.....	2.3	5.8	2.4	...	...	...	...
7/1/51.....	3.3	8.4	2.6	...	...	...	...
8/1/51.....	3.7	9.4	2.8	...	...	...	...
9/1/51.....	3.8	9.7	2.1	1.5	49.0	7.1	42.4
10/1/51.....	4.2	10.7	2.6	1.3	53.2	8.4	37.1
11/1/51.....	4.5	11.4	2.5	2.4	59.0	5.8	32.8
12/1/51.....	4.6	11.7	2.6	3.0	59.8	5.6	31.6
1/1/52.....	4.7	12.0	3.0	3.1	61.7	3.8	31.4
2/1/52.....	4.8	12.3	3.1	2.8	56.2	4.9	36.1
3/1/52.....	4.6	11.7	2.6	2.2	55.6	4.3	37.9
4/1/52.....	4.8	12.3	2.5	2.9	58.3	3.5	36.3

*Adapted from Kesterson and Hendrickson (1952).

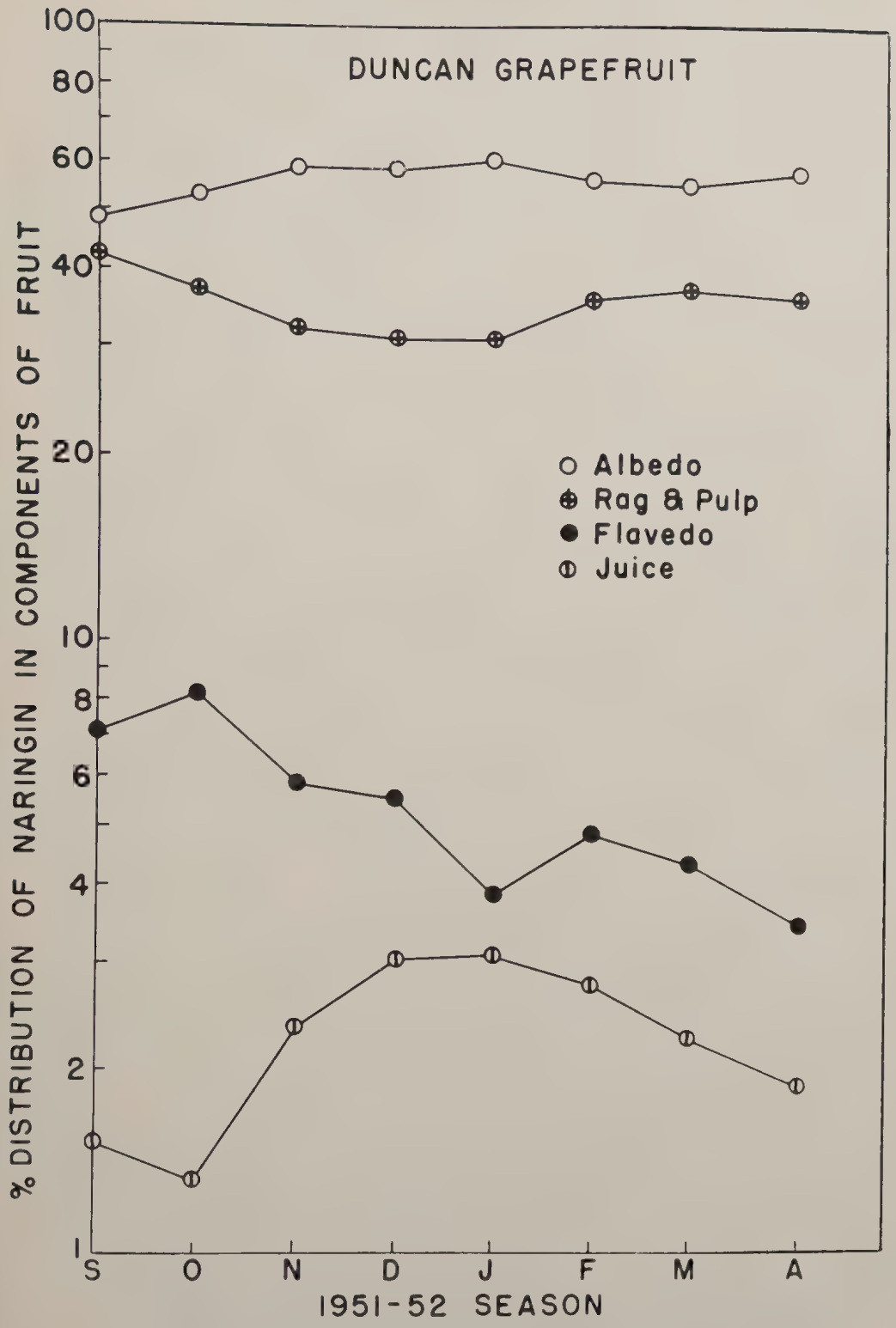


Fig. 33. Distribution of naringin in the component parts of Duncan grapefruit. (From Kesterson and Hendrickson, 1953.)

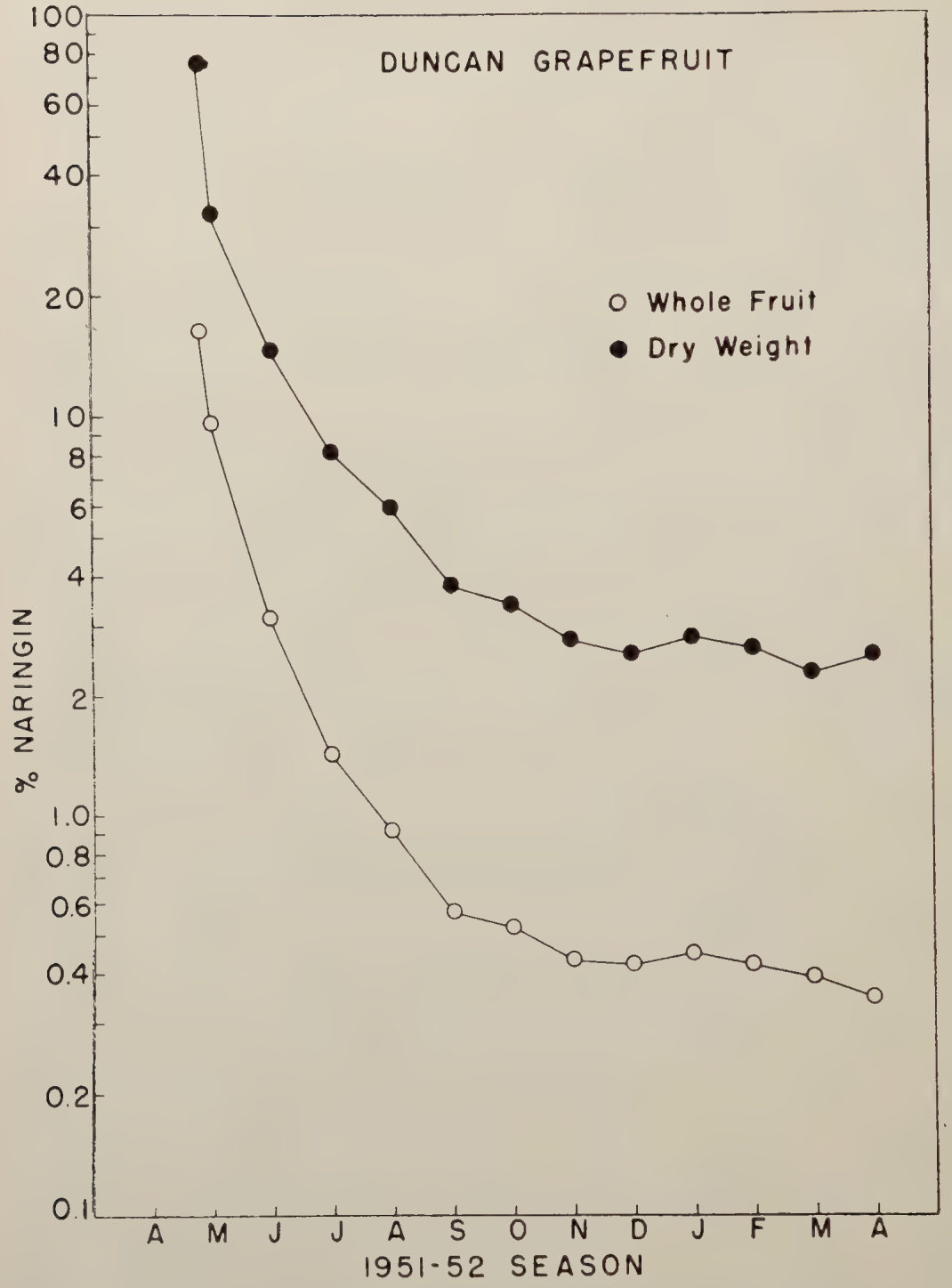


Fig. 34. Naringin content of Duncan grapefruit on both whole-fruit and dry-weight bases. (From Kesterson and Hendrickson, 1953.)

The total naringin content of different varieties of grapefruit, expressed as grams per fruit rather than percentage, is shown in figure 35. On this basis, the shaddock had the highest and the Thompson grapefruit had the lowest amount per whole fruit. It appears that the amount of naringin per fruit increased with the size of the fruit. If the naringin values are calculated as percentages of either dry weight or whole-fruit weight, Ruby (Ruby Red) had the most and shaddock had the least. [*Turn to page 144.*]

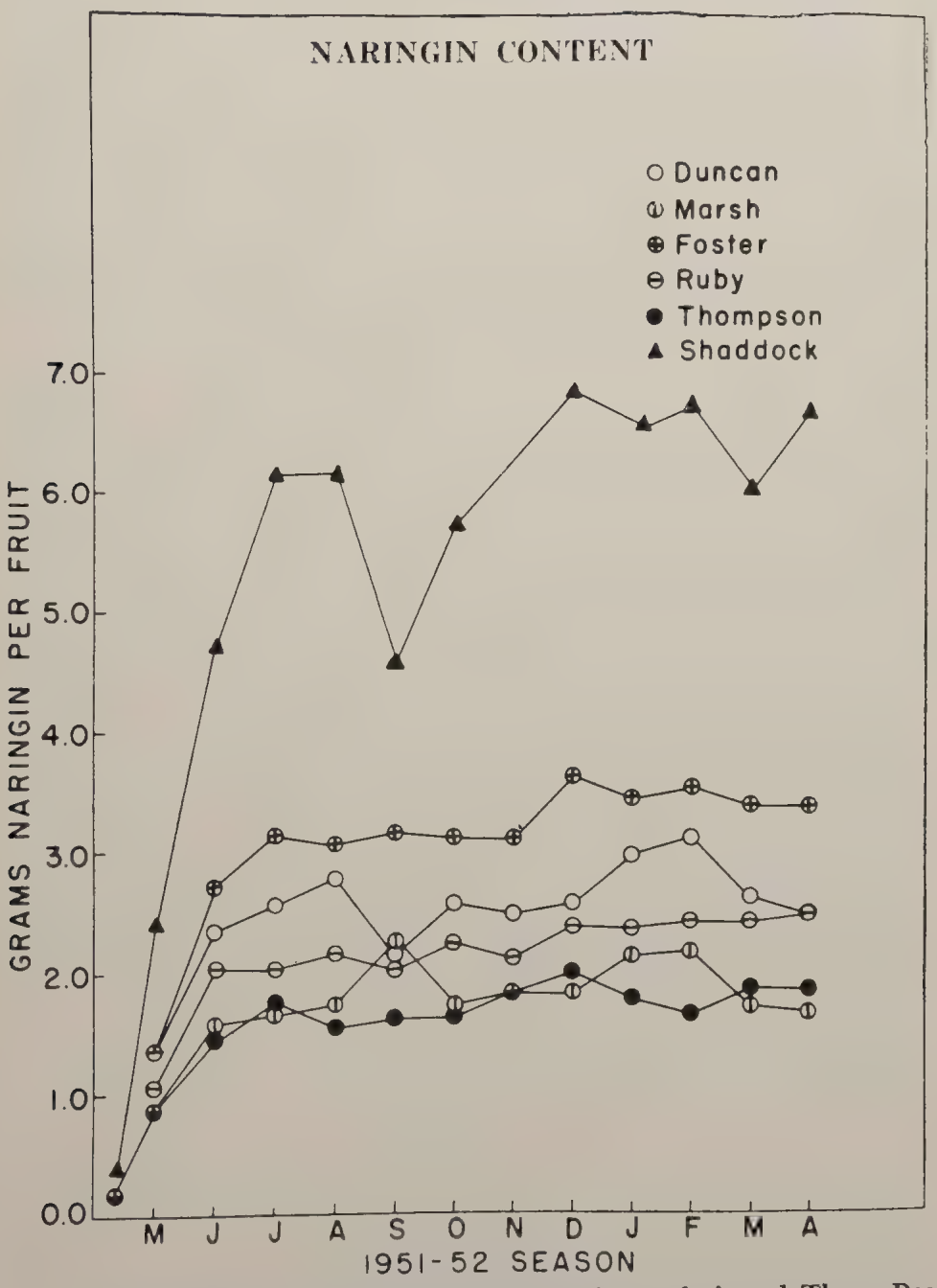


Fig. 35. Naringin content of 5 varieties of grapefruit and Thong Dee shaddock, on a per-fruit basis. (From Kesterson and Hendrickson, 1953.)

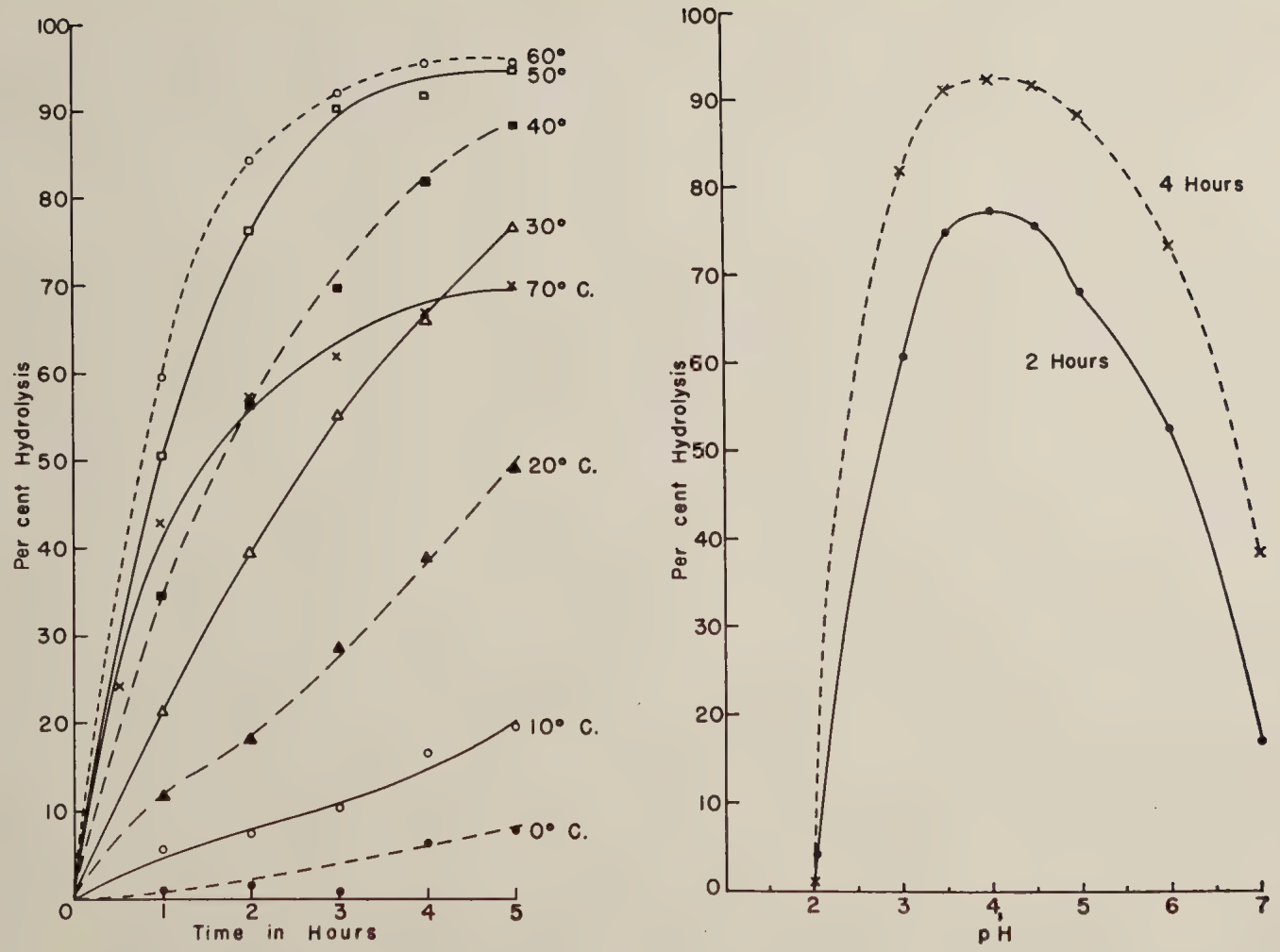


Fig. 36. Effects of temperature and pH on the rates of hydrolysis of naringin. (Refer to figure 37 for explanations of symbols.) (From Ting, 1958.)

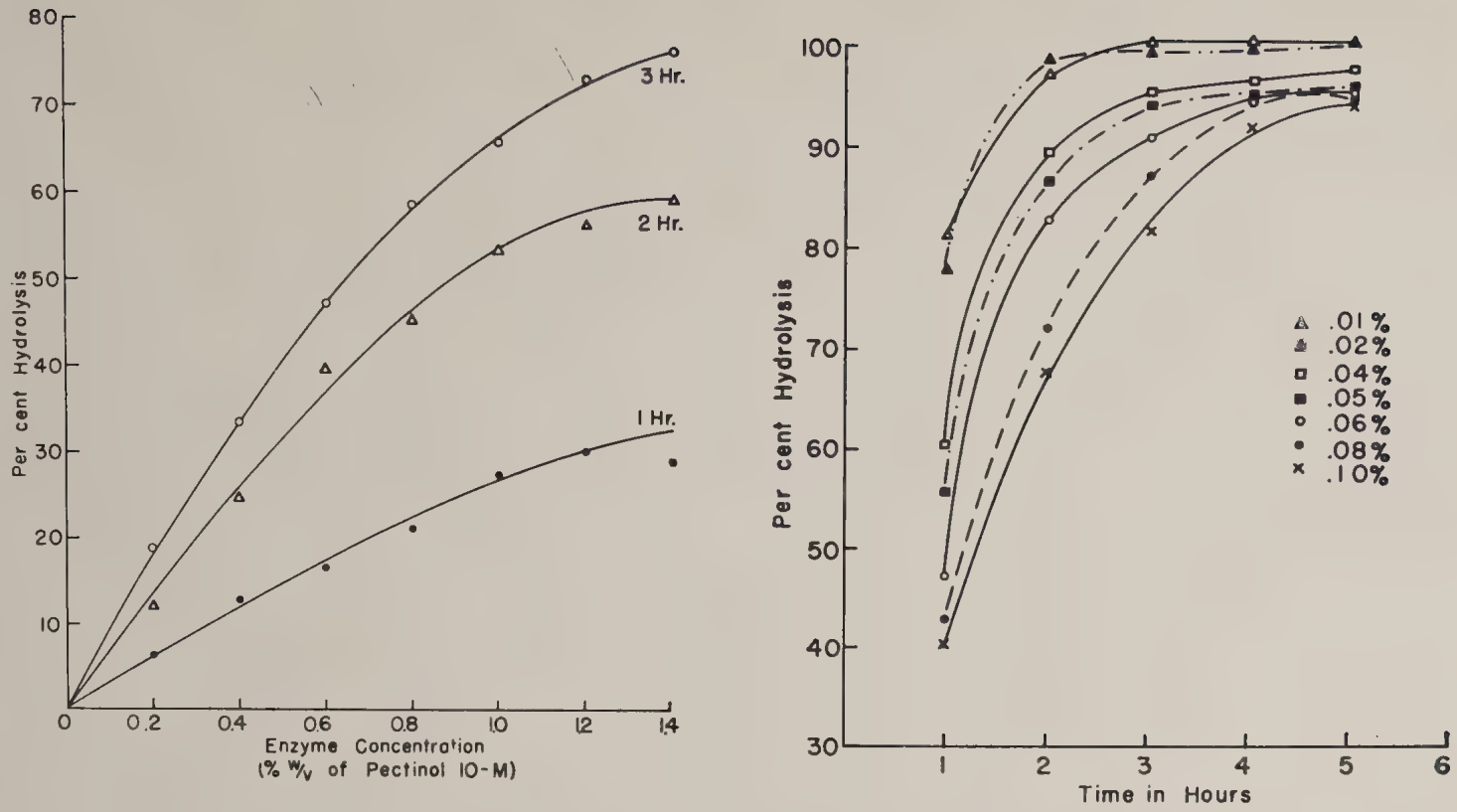


Fig. 37. Effects of concentrations of enzyme and substrate on the rates of hydrolysis of naringin. (From Ting, 1958.)

Lime *et al.* (1954) showed that the percentage of naringin in the edible portion of the Ruby (Ruby Red) grapefruit grown in Texas decreased as the fruit matured, but that the percentage curve leveled off somewhat at about the time the fruit was full grown. *(See table 84 of this chapter, page 187.)*

Enzymatic Hydrolysis of Naringin. Naringin is the constituent that imparts the bitter taste to grapefruit. Excessive bitterness in grapefruit products is not acceptable to consumers. Consequently, ways and means of debittering these commodities have received considerable study. Some progress was made on this problem when it was discovered that the enzymatic hydrolysis of naringin was highly correlated with the reduction of the bitter flavor.

Ting (1958) found, in certain commercial pectic enzyme preparations (Pectinol 10-M and Pectinol 100-D), an enzyme that would hydrolyze naringin to rhamnose, glucose, and naringenin. He used this material to remove the bitter flavor from grapefruit juice by the enzymatic hydrolysis of naringin. Before the enzyme naringinase in such preparations can be used in a practical way to reduce the bitterness in grapefruit juice, it will be essential to isolate the naringinase activity from the large quantities of pectic enzymes which readily clarify the juice.

The conditions that affected the rate of hydrolysis of naringin by Pectinol 10-M are shown in figures 36 and 37. The temperatures had a marked effect on the rate of hydrolysis, the maximum rate occurring at 60° C. Between 50 and 60°, hydrolysis was nearly complete in 5 hours. The activity curve of the enzyme was definitely reduced at 70° C. (122, 140, and 158° F.).

It is always useful and important to establish the pH range of activity of an enzyme. The maximum activity depends upon the pH and the ionic strength of the medium, as well as upon those properties of the medium which influence hydrogen bonding and hydration and solvation of the enzyme. The effect of pH on the hydrolysis of naringin by Pectinol is shown in figure 36. The maximum enzyme activity occurred at pH 4.0, but the activity differences were negligible within a range of pH 3.5 to 4.5.

Figure 37 shows the effect of the concentration of enzyme and substrate on the rate of hydrolysis of naringin. With respect to enzyme concentration, it is well known that the initial velocity of an enzyme reaction is directly proportional to the concentration of enzyme present. Ting's results showed that at the lower enzyme concentrations, the hydrolysis was proportional to the reaction time, while at higher concentrations, the rate of hydrolysis decreased with reaction time. This deviation from a linear relationship may have been caused by the presence of many complicating factors, including the presence of other enzymes, inhibitors, such as glucose, and the effects of end products.

The rates of hydrolysis of naringin by naringinase at different initial substrate concentrations are also shown in figure 37. The author calculated

the percentage of hydrolysis of the substrate by determining, at hourly intervals, the amounts of naringin in the reaction medium. Under these experimental conditions, more than 94 per cent of the original substrate (naringin) was hydrolyzed in 5 hours.

Griffiths and Lime (1959) reported on the factors affecting the use of the enzyme naringinase in reducing the bitterness of grapefruit pulp and products, as measured by the Davis test and taste evaluation. These researchers used an enzyme preparation called "Naringinase-C," an active preparation described by Thomas *et al.* (1958). Optimum conditions for enzymatic hydrolysis of naringin in grapefruit pulp and juice to prunin and naringenin were experimentally determined to be 50° C. (122° F.) and pH 3.10, at enzyme concentrations of .05–.01 per cent, and at incubation periods of 1 to 4 hours.

The data of Griffiths and Lime in table 69 show the relationship between the concentration of naringinase and the suspended solids, cloud, and bitterness. The last-named characteristic was indicated by the Davis color test and taste values of pasteurized grapefruit pulp held at 50° C. for various time

TABLE 69

EFFECT OF NARINGINASE CONCENTRATION AND TIME ON SUSPENDED SOLIDS, CLOUD, DAVIS TEST, AND BITTERNESS OF PASTEURIZED GRAPEFRUIT PULP HELD AT 50° C. (122° F.).*

Time (hours)	Enzyme (per cent)	Suspended solids (per cent)	Cloud (per cent transmission)	Naringin† (per cent)	Taste‡
0	Control	54	8.5	0.151	10+
10	Control	54	8.5	.155	10+
0.5	0.05	46	9.5	.147	7
1	.05	48	9.5	.114	4
2	.05	52	9.5	.089	1
4	.05	40	12.0	.049	1
0.5	.025	54	9.0	.151	8
1	.025	48	9.5	.147	5
2	.025	48	9.5	.111	3+
3	.025	47	9.0	.076	3
4	.025	46	9.5	.078	2
6	.025	44	10.5	.056	1
8	.025	42	22.0	.051	1
10	.025	40	38.5	.044	1
1	.01	52	9.0	.155	9
3	.01	50	9.5	.147	8
4	.01	52	9.5	.119	5
6	.01	46	9.5	.095	3
8	.01	48	9.5	.091	2
10	.01	46	10.0	.081	1

*From Griffiths and Lime (1959).
†Determined by the Davis test.
‡Code: 10, very bitter; 5, acceptable; 1, trace. Intermediate figures are proportional.

periods. They also noted that enzyme activity at a temperature of 4° C. (39° F.) for 44 hours hydrolyzed naringin to prunin without a corresponding decrease in the Davis colorimetric values. The data presented in the last 2 columns of the table do not seem to indicate that bitterness was highly correlated with the naringin values as determined by the Davis method.

The rate of enzymatic hydrolysis of naringin in grapefruit concentrate was studied by Olsen and Hill (1964). Their data in figure 38 illustrate the effects of enzyme concentration, storage temperature, and time on the rate of reaction of naringinase with naringin, resulting in a corresponding decrease in bitterness. An inspection of the data shows that naringin, and simultaneously the bitterness of the concentrate, decreased as the naringinase concentration, storage temperature, and time increased. In this study, the authors were able to reduce the naringin concentration in grapefruit concentrate as much as 88.9 per cent when the reaction was carried out with an .08 per cent naringinase solution held for 32 hours at 80° F. (27° C.). Bitterness in reconstituted juice was nearly eliminated when the naringin in the product was hydrolyzed with an .018 per cent naringinase solution and stored at 40° F. (4.4° C.) for 15 days. Naringinase activity was not detected in grapefruit concentrate (55° Brix) stored at –8° F. (–22° C.) over a 1-year period.

Pectic Substances

GRAPEFRUIT PEEL is the part ordinarily used in the commercial preparation of pectin. Therefore, most of the previously published reports on pectic substances in this fruit deal with amounts in the peel. Several of these reports are reviewed in chapter 3. That chapter also contains a discussion of the nomenclature of pectic substances and of the relationship of purity of pectin to methoxyl and polygalacturonic acid contents.

Pectic substances are composed primarily of polyanhydrogalacturonic acid units in unbranched chains which make up the basic framework of all pectic materials, together with different amounts of nongalacturonide carbohydrate materials, such as araban and galactan, which are probably in or closely associated with the molecule. The carboxyl groups of the molecule are usually esterified to about 80 per cent, and the remaining 20 per cent exist as free acids or salts. The secondary alcohol groups in some pectic substances are esterified as acetyl esters.

In addition to the differences in their composition, the pectic substances vary in molecular sizes.

It has been fairly well established that changes in the average molecular size of the pectin molecule are manifested by changes in fruit characteristics, such as hardness or softness, consistency and gel strength of processed citrus fruits, rate of drying, and subsequent rehydration. Since the basic monomer

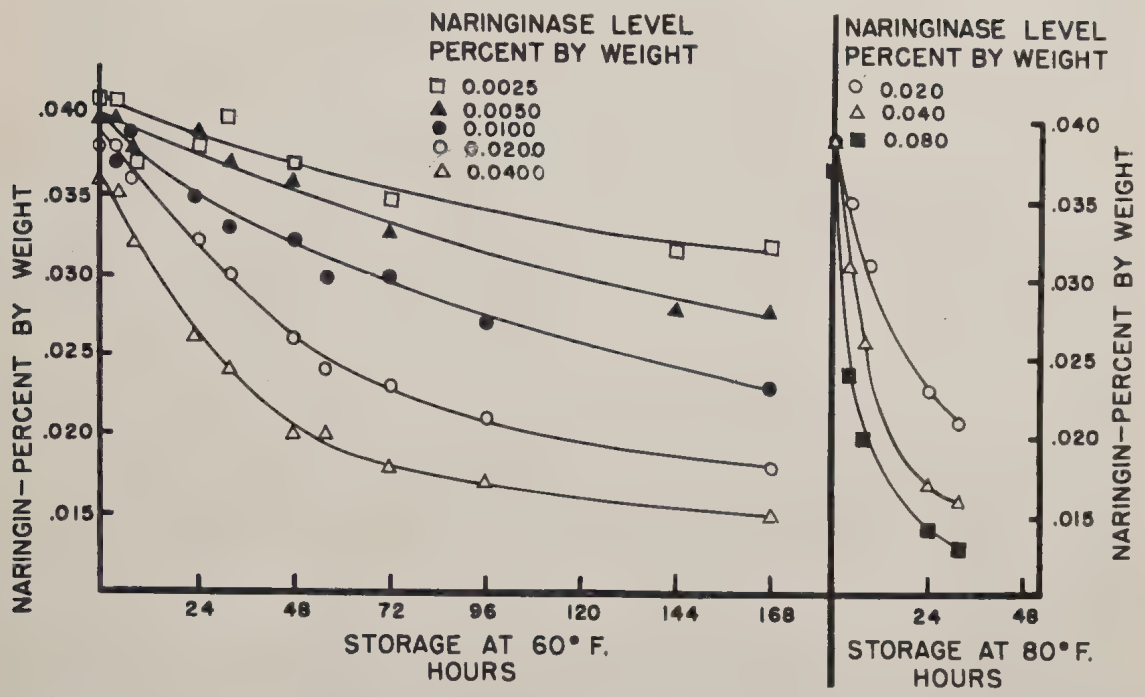


Fig. 38. Naringin contents in reconstituted grapefruit juices (10.5° Brix) after enzymatic debittering of grapefruit concentrates (55° Brix) during storage at 60 or 80° F. (16 or 27° C.), using different concentrations of naringinase. (From Olsen and Hill, 1964.)

of pectic substances is D-galacturonic acid, the increase in polymolecular size would also be reflected in chemical and physical characteristics of growing and maturing citrus fruits.

A considerable portion of the pectic substances of citrus fruits exists in tissues as protopectin. This fraction is defined as the water-insoluble parent pectic material which occurs in plant tissues. It follows, therefore, that the separation of protopectin from the various pectinic and pectic acids must be accomplished on the basis of differential solubility in water. A definition of protopectin based on its differential solubility in water is not precise, and examples can be cited to show that salts of pectic acid and low-ester pectinic acid will show a solubility similar to that of protopectin. In fact, it is impossible to reach a stage in the extraction of protopectin from plant tissues where no further soluble pectin is removed with the solvent. The best preparations of protopectin are usually obtained from fruit tissues, such as those of apples, lemons, oranges, and grapefruit, by removing all constituents that are soluble in water, alcohol, and ether. The material in the residues contains the cellulose A, hemicellulose A, hemicellulose B, pectin, protopectin, and protein. These fractions are designated and experimentally obtained by consecutive extractions with aqueous solutions of different hydrogen ion concentrations. The quantity of the various fractions that is obtained by such an extraction procedure depends upon the degree of susceptibility of the original material to degradation. The classification of the polysaccharides based on the solu-

bility of these fractions to overlap in various solvents has led to considerable confusion in the literature.

It has been well established experimentally that the polysaccharides vary considerably in the amount of uronides they contain. Those polysaccharides containing a high percentage of uronic acids are known as the pectic substances, and those with a low percentage of uronic acids are known as hemicelluloses. In some instances, the uronic acids occur frequently in the polysaccharide fraction, and such fractions are usually classed as hemicelluloses and not as pectic substances.

Sinclair and Crandall (1954) found pectin as calcium pectate to be 36.6 per cent of the alcohol-insoluble solids of juice vesicles, or 0.3 per cent of the fresh weight of pulp (average of 2 samples). As with a similar study on grapefruit peel (Sinclair and Crandall, 1949*a*, reviewed on page 226 ff. of chapter 3), the results were analyzed in detail to evaluate the accuracy of the method.

The juice vesicles of grapefruit were found to contain smaller amounts of alcohol-insoluble solids than the peel (compare table 70 with table 107 of chapter 3). On the dry-weight basis, these substances in the juice vesicles averaged 7.39 per cent. The soluble constituents, such as sugars, organic acids, most of the inorganic compounds, amino acids, essential oils, and glycosides, were extracted from the vesicles with 80 per cent alcohol. It is evident that most of the dry matter of the pulp was soluble in 80 per cent alcohol.

The carbon dioxide produced on hydrolyzing the alcohol-insoluble solids with 19 per cent hydrochloric acid was a measure of their total uronide content. The average amount was 7.08 per cent. The galacturonic acid anhydride equivalent may be calculated by multiplying the carbon dioxide values by 4.0; the average was 28.30 per cent. The value, as determined by titration of the total carboxyl groups, averaged 28.38 per cent.

The degree of esterification of the carboxyl groups was shown by the amounts of methoxyl reported as percentages of the alcohol-insoluble solids. The average was 3.40 per cent. Methoxyl equivalent to the total carbon dioxide would be 4.98 per cent. Therefore, the percentage of methylation was $3.40/4.98 \times 100 = 68.27$ per cent.

The total carbon dioxide of a given fraction was equal to the sum of the esterified and nonesterified carboxyl groups, expressed as carbon dioxide (table 70). The nonesterified groups represent the carboxyl groups combined with the inorganic cations. The relatively low methoxyl content and the absence of free acidity indicate that some of the carboxyl groups were combined with the cations of the alcohol-insoluble fraction. This can be shown experimentally by converting the methoxyl values to equivalent percentages of carbon dioxide plus the carbon dioxide calculated from the free acidity of the ash-free alcohol-insoluble solids. The esterified carbon dioxide aver-

TABLE 70

CARBON DIOXIDE AND METHOXYL CONTENTS OF ALCOHOL-INSOLUBLE SOLIDS (A.I.S.) OF VESICLES OF GRAPEFRUIT PULP IN RELATION TO THEIR EQUIVALENTS OF NONESTERIFIED AND ESTERIFIED CARBOXYL GROUPS.*

Sample numbers	Moisture (per cent fresh weight)	Alcohol-insoluble solids		CO_2 produced on hydrolysis with 19 per cent HCl		Methoxyl (per cent A.I.S.)	Galacturonic anhydride		Carboxyl groups (as CO_2)			Non-esterified —COOH in A.I.S. (meq/gram)	Calcium in A.I.S. (meq/gram)	Alkaline ash in A.I.S. (meq/gram)
		(per cent dry weight)	(per cent fresh weight)	(per cent dry weight)	(per cent A.I.S.)†		$CO_2 \times 4$ (per cent A.I.S.)	Equivalent to total carboxyl groups (per cent A.I.S.)	Non-esterified (per cent A.I.S.)	Esterified (per cent A.I.S.)	Totals (per cent A.I.S.)			
1.....	89.63	7.62	0.79	0.54	7.03	3.35	28.12	28.04	2.26	4.75	7.01	0.51	0.59	0.74
2.....	88.96	7.16	.79	.51	7.12	3.44	28.48	28.72	2.30	4.88	7.18	.53	.61	.73
Averages..	89.30	7.39	.79	.52	7.08	3.40	28.30	28.38	2.28	4.82	7.10	.52	.60	.74

*From Sinclair and Crandall (1954).
†Alcohol-insoluble solids, calculated on moisture-free basis.

aged 4.82 per cent, while that equivalent to the free acidity averaged 2.28 per cent. The sum of the 2 fractions (7.10 per cent) was in good agreement with the 7.08 per cent determined by hydrolysis with 19 per cent hydrochloric acid.

The data of table 71 show the relationship between the methoxyl, galacturonic acid, and carboxyl groups of the alcohol-insoluble fraction of juice vesicles. The galacturonic acid values were determined by multiplying the percentages of carbon dioxide by 4.411. In addition to the methoxyl values experimentally determined on the alcohol-insoluble fraction, the methoxyl values equivalent to the total carboxyl groups were recorded. The degree of esterification of the galacturonic acid in the alcohol-insoluble fractions is shown by the ratio of methoxyl to galacturonic (0.109).

In terms of equivalent methoxyl, the ratio of total carboxyl groups to galacturonic acid shows the greatest amount of methoxyl that can occur in the polygalacturonide of the alcohol-insoluble fraction. The average ratio was 0.160. This value agrees satisfactorily with the theoretical value of 16.32 per cent for a completely esterified polygalacturonide. The close agreement between theoretical and experimental values is evidence that the total carboxyl groups were derived from the pectin fraction of the alcohol-insoluble solids.

The percentages of the total carboxyl groups esterified are reported in the last column of table 71. The average of 67.90 per cent agrees closely with the value of 68.27 per cent calculated from the methoxyl percentages in table 70.

The data of table 72 show the relationship of the amount of uronic acid anhydride to the amount of furfural in the alcohol-insoluble solids. Uronic acid anhydride determinations were calculated from both the carbon dioxide and the saponification values of the alcohol-insoluble solids. The values calculated from the percentages of carbon dioxide were much higher than those calculated from the saponification values. The lower values can be partly explained by the fact that some of the pectin was combined with inorganic cations through the carboxyl groups; this signifies that not all the carboxyl groups were completely methoxylated. The values for the uronic acid anhydride were obtained by multiplying the percentages of carbon dioxide in the alcohol-insoluble solids by the factor 4.0. The percentages of total furfural equivalent to the uronic acid anhydride varied considerably in the different samples. The percentages of furfural derived from substances other than uronic acid, when multiplied by the factor 1.72, gave the pentosan values of the alcohol-insoluble solids. The pentosan values may be expressed as pentoses if the furfural values are multiplied by the factor 1.95 instead of 1.72. The mean furfural yielded by the alcohol-insoluble fractions of the vesicles of grapefruit was 12.96 per cent.

TABLE 71

RELATIONSHIPS BETWEEN METHOXYL, GALACTURONIC ACID, AND CARBOXYL GROUPS OF ALCOHOL-INSOLUBLE SOLIDS (A.I.S.) OF GRAPEFRUIT VESICLES.*

Sample numbers	Total CO_2 (per cent of A.I.S.)	Per cent galacturonic acid in A.I.S. ($CO_2 \times 4.411$)	Methoxyl		Ratios of methoxyl to galacturonic acid in A.I.S.	Ratios of total carboxyl groups to galacturonic acid in A.I.S. (as equivalent methoxyl)	Polygalacturonic acid esterified (per cent)
			Per cent of A.I.S.	Equivalent to total carboxyl groups (per cent of A.I.S.)			
1	7.03	31.01	3.35	4.94	0.108	0.159	67.81
2	7.12	31.41	3.44	5.06	0.110	0.161	67.98
Averages	7.08	31.21	3.40	5.00	0.109	0.160	67.90

*From Sinclair and Crandall (1954).

TABLE 72

RELATIONSHIP OF URONIC ACID ANHYDRIDE TO FURFURAL CONTENTS OF ALCOHOL-INSOLUBLE SOLIDS (A.I.S.) OF GRAPEFRUIT VESICLES.*

Sample numbers	Total CO_2 (per cent of A.I.S.)	Uronic acid (galacturonic) anhydrides (per cent of A.I.S.)		Furfural (per cent of A.I.S.)			Pentosans, equivalent to furfural from substances other than uronic acid anhydrides $\times$ 1.72 (per cent of A.I.S.)
		Equivalent to $CO_2 \times 4$	Equivalent to methoxyl $\times$ 5.677	Equivalent to uronic acid anhydrides $\times$ 0.235	Totals determined	From substances other than uronic acid anhydrides	
1	7.03	28.12	19.02	6.61	13.12	6.51	11.20
2	7.12	28.48	19.53	6.69	12.81	6.12	10.53
Averages	7.08	28.30	19.28	6.65	12.96	6.32	10.86

*From Sinclair and Crandall (1954).

The yield of furfural from citrus pulp is derived chiefly from the pentoses of the pentosans and from the uronic acids and pentoses of the pectic substances. In plant materials, the uronic acids occur chiefly in pectic materials and hemicelluloses; in citrus pulp, however, the total uronic acid content is highly and positively correlated with the total amount of pectic material isolated. The furfural obtained by the difference between the total furfural of the vesicles and the amount equivalent to the uronic acids is ordinarily used to calculate the pentosans. On hydrolysis with 19 per cent hydrochloric acid, uronic acids yield a quantitative amount of carbon dioxide, but the furfural produced is less than the theoretical amount.

Lefèvre and Tollens (1907) found that glucuronic acid anhydride, on hydrolysis with 12 per cent hydrochloric acid, yielded, on the average, 19.11 per cent of its weight as furfural, or a ratio of furfural to glucuronic acid anhydride of 1:5.23. Similar determinations were made by Norris and Resch (1935), who found that glucuronic acid anhydride (euxanthic acid) yielded 21.48 per cent of its weight as furfural, or a ratio of furfural to glucuronic acid anhydride of 1:4.66. With galacturonic acid anhydride, the yield of furfural was 23.50 per cent, or a ratio of furfural to galacturonic acid anhydride of 1:4.26. The percentage of furfural derived from galacturonic acid anhydride was equal to the latter divided by 4.26. This relation gave a close approximation of the amount of furfural yielded by the uronic acids of the alcohol-insoluble solids. Substances other than uronic acids and polyuronides that would yield furfural, such as pentoses and hexoses, were not present because they had been extracted from the pulp with 80 per cent alcohol.

The water-soluble and acid-soluble pectic substances of alcohol-insoluble fractions of grapefruit vesicles are reported as calcium pectate in table 73, based on data developed by Sinclair and Crandall (1954). Although hot water did not extract so much pectin as did dilute hydrochloric acid solution, the calcium pectate obtained from the aqueous extract was a better product and more easily purified than that from the acid extract. The calcium pectates from the aqueous extracts contained higher percentages of carbon dioxide and calcium than those from the acid extracts. The furfural values, on the other hand, were slightly larger in the acid-extracted pectin than in that extracted by water.

The degree of purity of the calcium pectates can also be observed from the ratios of calcium to carbon dioxide. The theoretical ratio of calcium to carbon dioxide is 0.455. The ratios from the water-soluble pectin showed satisfactory agreement with the theoretical value. The ratios from the acid-extracted pectin were high, owing to the relatively low percentages of carbon dioxide.

As shown in the table, the total calcium pectate obtained was the sum of that determined in the aqueous and acid extracts. It amounted to 36.10 per cent of the alcohol-insoluble fraction. The amount of carbon dioxide equiva-

lent to the total calcium pectate was 5.92 per cent. This was 83.75 per cent of the total carbon dioxide. The sum of the carbon dioxide in the total calcium pectate and in the residue was 6.44 per cent. Because the total carbon dioxide determined by hydrolysis of the alcohol-insoluble solids was 7.08 per cent, this left an undetermined fraction of 0.64 per cent (mean), representing the amount lost in the extraction and precipitation procedures.

The data in table 74 show the degree of efficiency with which the calcium ion precipitated the pectic substances from the aqueous and acid extracts of the various alcohol-insoluble fractions. The mean amount of carbon dioxide recovered as calcium pectate from aqueous extracts was 84.74 per cent; from the acid extracts, it was 95.70 per cent. Undoubtedly, a portion of the loss in carbon dioxide occurred in the large volume of wash water used to free the calcium pectate of chlorides.

The relationship of the carbon dioxide of the alcohol-insoluble solids to the sum of the various fractions is also shown in table 74. The sum of the total carbon dioxide in the aqueous extract, acid extract, and residue was 99.08 per cent of the total carbon dioxide, or a loss of less than 1 per cent in the extraction process. The material that remained after extraction of the pectins from the alcohol-insoluble solids was composed chiefly of cellulose and hemicellulose, which in turn contained a comparatively small amount of firmly bound pectic material that was difficult to extract and determine quantitatively. The extractants used to dissolve the pectin also dissolved

TABLE 73

WATER-SOLUBLE AND ACID-SOLUBLE PECTIN OF ALCOHOL-INSOLUBLE SOLIDS (A.I.S.) OF GRAPEFRUIT VESICLES, DETERMINED AS CALCIUM PECTATE.*

Sample numbers	Total CO_2 (per cent of A.I.S.)	Water-soluble pectin as calcium pectate					Total calcium pectate determined (per cent of A.I.S.)	CO_2 in calcium pectate (per cent of A.I.S.)
		Total (per cent of A.I.S.)	Constituents, percentages of:			Ratios of Ca to CO_2		
			CO_2	Calcium	Furfural			
1	7.03	10.91	19.14	8.40	18.19	0.439	36.56	5.96
2	7.12	12.66	18.54	8.45	18.72	0.456	36.63	5.89
Averages	7.08	11.78	18.84	8.42	18.46	0.448	36.60	5.92

Sample numbers	Acid-soluble pectin as calcium pectate					Calcium pectate calculated from CO_2 (per cent)	CO_2 in residue (per cent of A.I.S.)	Sums of CO_2 in calcium pectate and residue (per cent of A.I.S.)	Undetermined CO_2 (per cent of A.I.S.)
	Total (per cent of A.I.S.)	Constituents, percentages of:			Ratios of Ca to CO_2				
		CO_2	Calcium	Furfural					
1	25.65	15.08	7.55	19.24	0.501	84.78	0.54	6.50	0.53
2	23.97	14.79	7.09	19.01	0.479	82.72	.49	6.38	.74
Averages	24.81	14.94	7.32	19.12	0.490	83.75	.52	6.44	.64

*From Sinclair and Crandall (1954).

TABLE 74

BALANCE SHEET SHOWING RELATIONSHIP OF CARBON DIOXIDE IN ALCOHOL-INSOLUBLE SOLIDS (A.I.S.) TO AMOUNTS DETERMINED IN CALCIUM PECTATE FROM AQUEOUS AND ACID EXTRACTS OF GRAPEFRUIT VESICLES.*

Sample numbers	Total CO_2 (per cent of A.I.S.)	CO_2 in aqueous extract			CO_2 in acid extract			CO_2 in residue (per cent of A.I.S.)	Sums of CO_2 fractions determined (per cent of A.I.S.)	Percentages of total CO_2 determined
		Total (per cent of A.I.S.)	Equivalent to Ca pectate in extract (per cent of A.I.S.)	Percentage accounted for	Total (per cent of A.I.S.)	Equivalent to Ca pectate in extract (per cent of A.I.S.)	Percentage accounted for			
1	7.03	2.41	2.09	86.72	3.98	3.87	97.24	0.54	6.93	98.58
2	7.12	2.84	2.35	82.75	3.76	3.54	94.15	.49	7.09	99.58
Averages	7.08	2.62	2.22	84.74	3.87	3.70	95.70	.52	7.01	99.08

*From Sinclair and Crandall (1954).

various portions of the hemicellulose. This condition made it difficult to extract and precipitate all the uronic acid carbon dioxide as calcium pectate. The quantity of carbon dioxide in the residues of grapefruit vesicles was 0.52 per cent (mean value) of the total alcohol-insoluble solids.

The results of Anderson (1927) appeared to indicate that the pectic substances sometimes permeated the intermicellar spaces of the cellulose portions of the cell wall, and by chemically removing the cellulose material, left the thickness of the cell wall unchanged.

Seasonal Changes in the Pectic Constituents of Grapefruit. In studying seasonal changes in the composition of Silver Cluster grapefruit, Rouse *et al.* (1965) picked monthly samples from the same trees from August through May during both the 1961–62 and 1962–63 seasons, and determined the various pectic fractions in the component parts of the fruit. The pectic substances were presented in 3 separate fractions: water-soluble, 0.5 per cent ammonium oxalate-soluble, and .05 *N* sodium hydroxide-soluble. The anhydrogalacturonic acid (AGA) values were determined on each fraction, and the results were reported as the percentages of pectic substances on a dry-weight basis.

In the peel tissues, the total pectic substances, as indicated by the sum of the 3 pectic fractions, were lowest in September (10 per cent) and highest in February (15.4 per cent), as shown by figure 39. For comparative purposes, the water-soluble pectic substances ranged in value from a low of 1 per cent in September to a high of 2.7 per cent in February. The concentration of ammonium oxalate-soluble pectic material showed a range of variation of approximately 1.4 per cent throughout the 10-month sampling period. The pectic substances extractable with .05 *N* sodium hydroxide ranged from a minimum value of 1 per cent in September to a maximum of 4.8 per cent in March, followed by a decline to 2 per cent in May.

The segment membrane tissue (endocarp tissue enclosing the juice vesicles) contained a greater quantity of total pectic substances than the other tissue components reported in the paper (figure 40). These substances showed a total seasonal increase during the growth period of August through May, ranging from 16 per cent to about 23 per cent. The curves for both ammonium oxalate- and sodium hydroxide-soluble pectic materials had a general upward trend during the second half of the period, although the alkali-soluble pectic substances decreased in the May sample.

The total pectic substances in the juice vesicles ranged, during the 10-month sampling period, from 7.2 to 13.2 per cent of their dry weights (figure 41). The curves representing the 3 pectic fractions of the juice vesicles show rates of increase in pectic constituents similar to those of the membrane tissue. The authors drew attention to the fact that the freeze of 1962–63 increased the quantity of ammonium oxalate-soluble pectic material (2.2 to 8.4 per cent) above that of the normal 1961–62 season (1.9 to 4.4 per cent). The seasonal

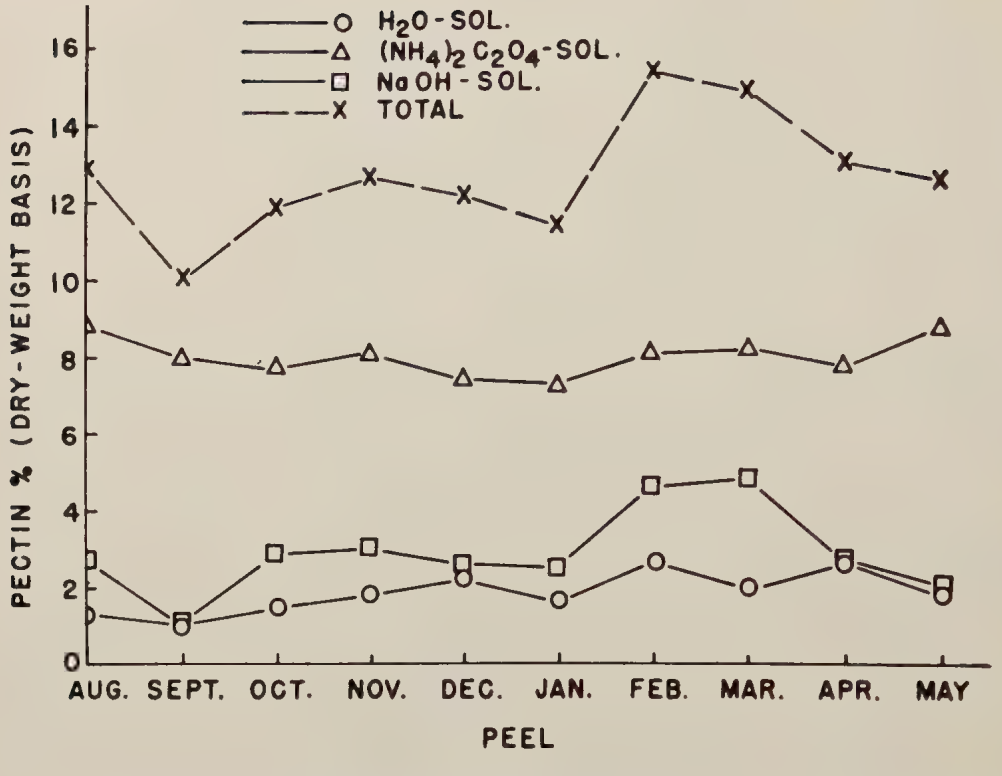


Fig. 39. Changes in the pectic fractions of the peel during maturation of Silver Cluster grapefruit. Each curve represents the mean values for the 1961–62 and 1962–63 seasons. (From Rouse *et al.*, 1965.)

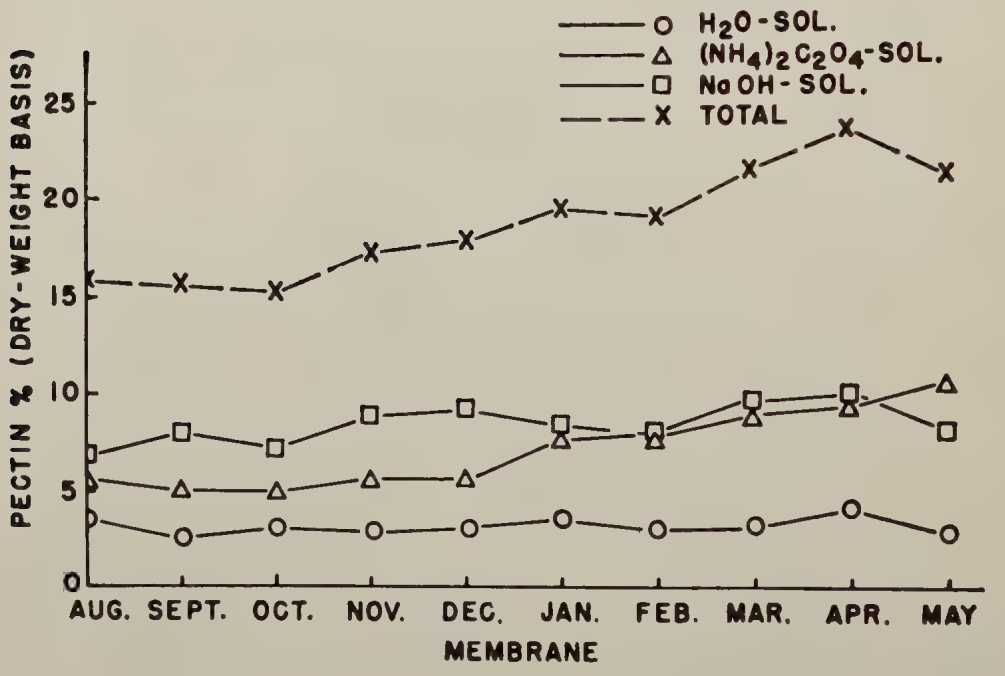


Fig. 40. Changes in the pectic fractions of the membrane tissues during maturation of Silver Cluster grapefruit. Each curve represents the mean values for the 1961–62 and 1962–63 seasons. (From Rouse *et al.*, 1965.)

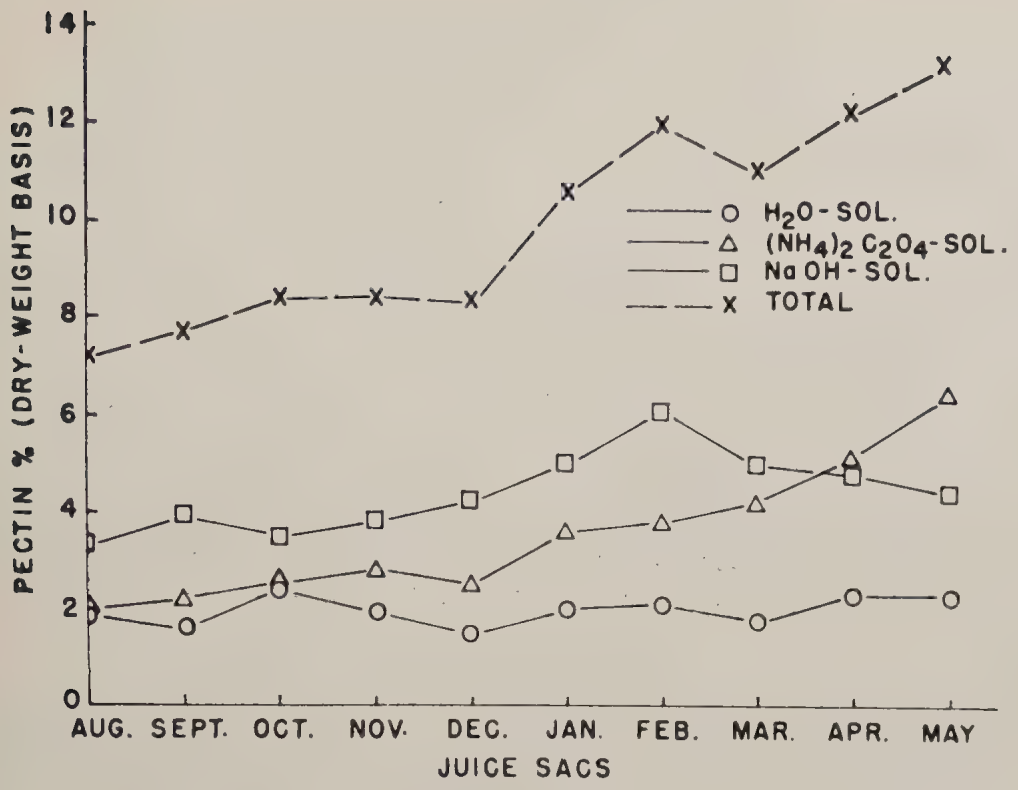


Fig. 41. Changes in the pectic fractions of the juice vesicles during maturation of Silver Cluster grapefruit. Each curve represents the mean values for the 1961–62 and 1962–63 seasons. (From Rouse *et al.*, 1965.)

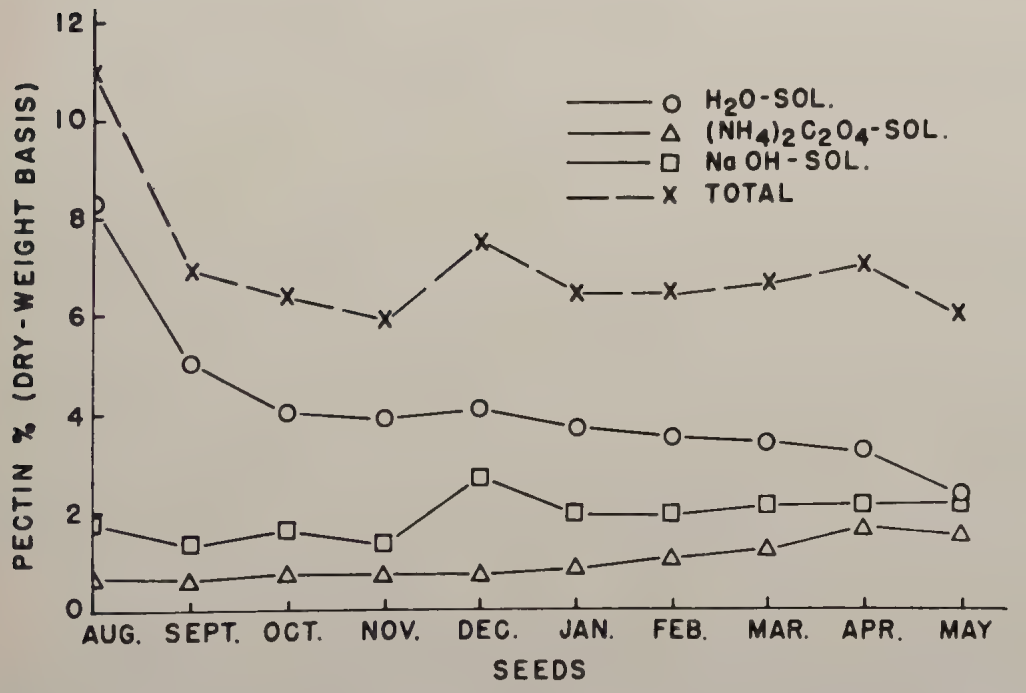


Fig. 42. Changes in the pectic fractions of the seeds during maturation of Silver Cluster grapefruit. Each curve represents the mean values for the 1961–62 and 1962–63 seasons. (From Rouse *et al.*, 1965.)

changes in concentrations of the ammonium oxalate-soluble pectic fraction during the 2-year sampling period are graphically demonstrated in the curve shown in figure 41.

The seeds of Silver Cluster grapefruit contained a variable quantity of pectic substances (figure 42). In the initial sample (August), the water-soluble pectic fraction amounted to nearly 75 per cent of the total pectic materials. During the sampling period of August through May, the water-soluble pectic fraction decreased from 8.3 to 2.3 per cent, a total reduction of about 72 per cent. The ammonium oxalate- and sodium hydroxide-soluble fractions amounted to less than 2 per cent of the dry weight in the initial sample, and less than 3 per cent in the final sample. Since the pectic contents of the tissues were reported on a dry-weight basis, the significant seasonal reduction in the water-soluble pectic material of the seeds was inversely related to the increase in dry weight, which varied from 26.6 to 48 per cent during the growth cycle.

Enzymes

THE ENZYMES found in citrus are much the same as those found in other green plants, and therefore may be classed under the usual headings, such as hydrolyzing, oxidizing, fermenting, and probably, to a lesser extent, coagulating or clotting enzymes. The effects of some of these enzymes in citrus tissues have been studied, but relatively few have been isolated. The present discussion concerns only those that have actually been isolated from grapefruit, and the activity of pectinesterase, which is of importance in the commercial processing of the juice. Glycosidase, which has been isolated from the peel, is briefly discussed on page 274 of chapter 3. For a general discussion of enzymes in citrus fruits, see the chapter on enzymes and enzyme activity by Joslyn and Pilnik (1961).

Enzymes are proteins of high molecular weight whose function is to accelerate and catalyze the rate of a particular chemical reaction. In general, there is a specific type of enzyme for each type of metabolic reaction. In some instances, an enzyme may show specificity for a particular chemical linkage; other enzymes are similarly specific for catalyzing a given compound or substrate. Since there are a great many different chemical reactions which occur in living tissues, there are a great many enzymes in each cell. Hence, the activity of enzymes is the mechanism by means of which metabolic reactions are performed in biological systems.

Although some enzymes are composed entirely of proteins of high molecular weight, there are others which are composed of 2 fractions, a protein fraction and a nonprotein prosthetic group called *coenzymes*. Prosthetic groups can consist of single metal atoms, such as copper, manganese, magnesium, and iron, as well as complex organic compounds, such as the water-

soluble vitamins. Therefore, the enzymes of higher plants and vitamins of the B-complex, such as biotin, nicotinic acid, pantothenic acid, thiamin, folic acid, riboflavin (vitamin B_2) and pyridoxine (vitamin B_6), constitute the reactive portion of 1 or more coenzymes. If nicotinic acid and adenine appear together in phosphopyridine nucleotide, this prosthetic group can unite with any one of several proteins to produce a variety of different enzymes, all of which react to produce hydrogen from oxidizable substances. The two coenzyme forms of nicotinic acid, diphosphopyridine nucleotide (DPN) and triphosphopyridine nucleotide (TPN), react specifically to transfer hydrogen in a variety of biological reactions.

The energy that molecules must possess before they are capable of reacting is called the *energy of activation.* It has been established that enzymes have a marked tendency to decrease the energy of activation (also known as the *critical increment*) necessary for reaction. Because of the lowered energy of activation, a larger number of the reacting molecules attain the necessary energy per unit time, thus increasing the rate of reaction. The chemical reactions of biological systems occur at accelerated rates because of the decreased activation energies involved in enzymatic changes. It is well known that the catalytic hydrolysis of sucrose by hydrogen ions requires an energy of activation of 25,560 cal for reaction; but if the reaction is carried out by the enzyme invertase, the energy of activation is reduced to approximately 9,000 cal. The numerous chemical reactions of most biological tissues would proceed at very low rates in the absence of cellular enzymes. The presence of specific enzymes (or catalysts) in the cells increases the rate of chemical reactions without their being significantly changed or destroyed during the cycle.

Phosphatase. Axelrod (1947) was the first to isolate an enzyme from citrus juice; he found phosphatase in navel and Valencia oranges, Eureka lemons, and Marsh grapefruit. Whole juice contained 5.32 PNP per milliliter, 1 PNP unit being that quantity of enzyme which will liberate 1 γ of nitrophenol per minute per 7 milliliters of reaction mixture at 37.8° C. (100° F.). There was much more phosphatase in the peel tissues; see chapter 3, under "Enzymes in Peel and Seeds."

Peroxidase. Davis (1942) isolated and measured the amounts of peroxidase in tissues of navel orange, Marsh grapefruit, lemon, and tangerine. He found 0.7 and 14.6 peroxidase units (PU) in grapefruit juice sediment and endocarp, respectively, which was only a small fraction of the amounts in the outer flavedo and inner seed coat.

Peroxidase, in common with catalase, contains iron porphyrin compounds as its prosthetic groups. This enzyme has the capacity to oxidize a number of substrates, using hydrogen peroxide as the oxidant. Comparisons of the peroxidase activities of different types of citrus fruits are shown in figure 43 (Vines and Oberbacher, 1962). The peroxidase activity was greatest in the

immature small fruit, and the enzyme activity, on the basis of fresh weight, rapidly decreased with an increase in fruit diameter up to 7 centimeters. Marsh Seedless grapefruit had the highest concentration of peroxidase activity in fruits with diameters between 2 and 4 centimeters. As the fruit increased in size, the peroxidase activity was concentrated in the flavedo and disappeared from the albedo and juice. Thus, the total peroxidase activity per fruit increased during its growth period, but if the experimental results were to be expressed on a fresh-weight basis, the concentration of the enzyme decreased with increase in fruit size.

Ascorbic Acid Oxidase. Frankenthal (1939) noted the presence of ascorbic acid oxidase in citrus peel on the basis of the methylene blue reducing system. This postulation was based chiefly on the fact that when peel juice was added to pulp juice, the ascorbic acid disappeared from the blend. Huelin and Stephens (1948) found significant quantities of ascorbic acid oxidase in orange

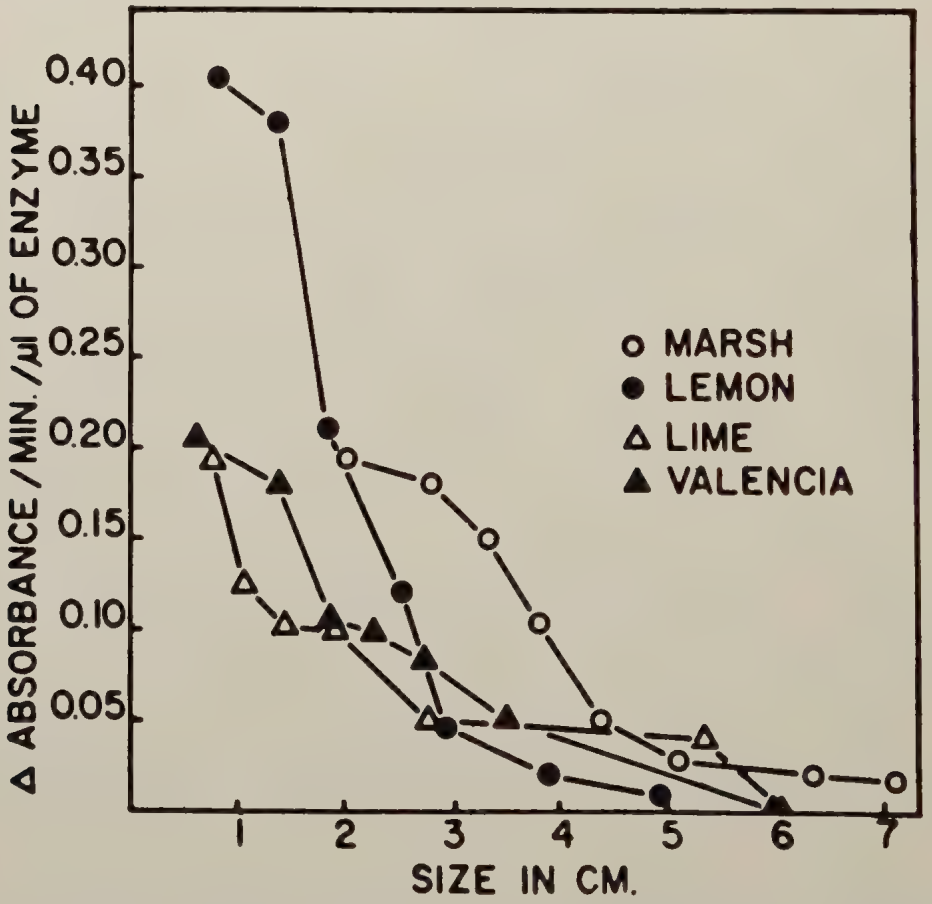


Fig. 43. Peroxidase: A comparison of the enzyme activity among several citrus fruits, ranging in diameter from less than 1 centimeter to 7 centimeters. Enzyme activity is expressed as the rate of change in absorbance per minute at 470 millimicrons per microliter, with hydrogen peroxide as the substrate and o-phenylenediamine as the indicator. (From Vines and Oberbacher, 1962.)

peel, but only negligible amounts in the juice. It is well known that many substances can oxidize ascorbic acid in the absence of ascorbic acid oxidase.

Vines and Oberbacher (1962, 1963), in order to establish criteria showing that ascorbic acid oxidase existed in various citrus fruits, experimentally determined its presence in immature fruits, and found that the enzyme was specific for ascorbic acid, had a stoichiometric relationship with the substrate and oxygen uptake, was inhibited by cyanide and diethyldithiocarbamate, and was oxygen-dependent. The authors determined that the optimum activity occurred at pH 5.6.

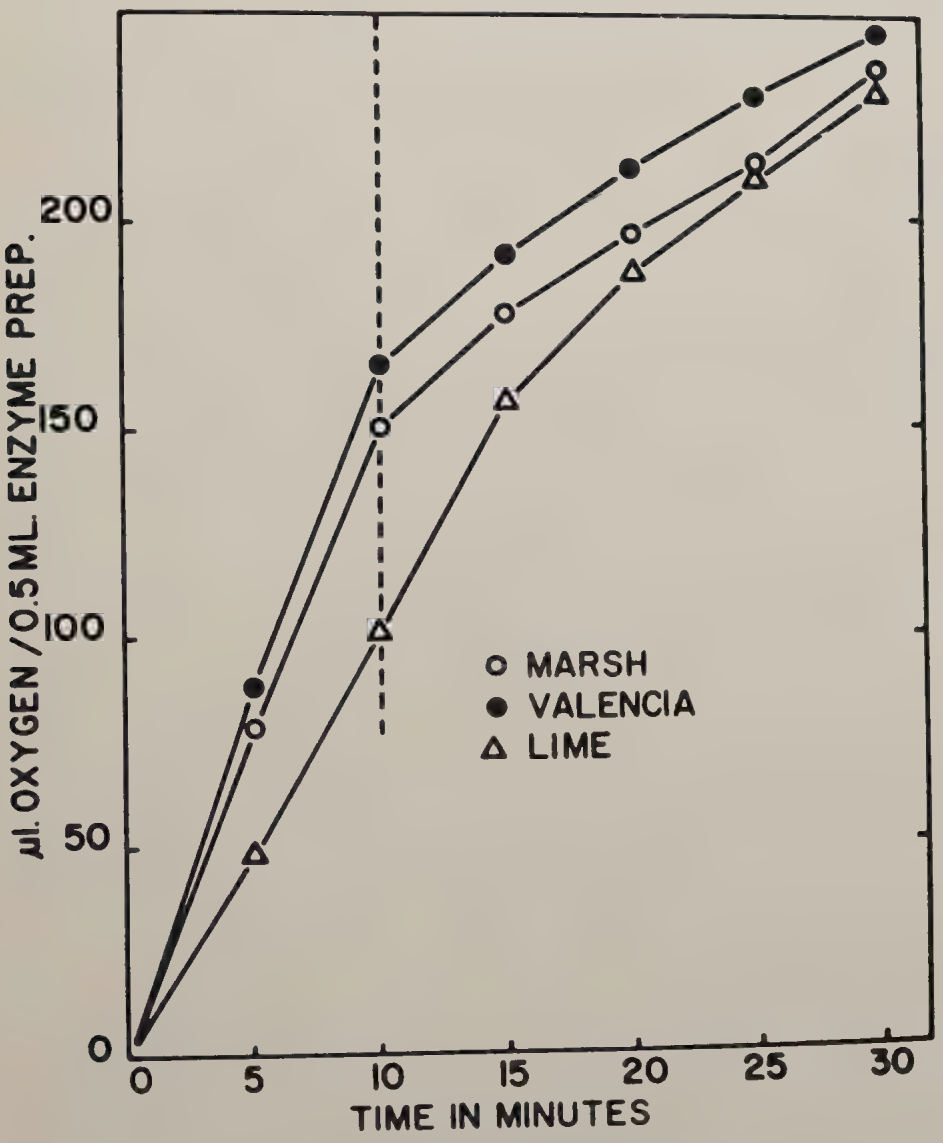


Fig. 44. Oxygen uptake of enzyme preparations from 3 citrus fruits (whole). The reaction vessels contained 30 micromoles of ascorbic acid and 0.2 milliliter of enzyme preparation in a total of 3 milliliters of 0.1 M phosphate buffer at pH 5.6. (From Vines and Oberbacher, 1962.)

The oxygen uptake of ascorbic acid oxidase from whole Marsh Seedless grapefruit and other commercial citrus varieties is shown in figure 44, from Vines and Oberbacher (1962). Their absorbance data were manometrically converted to enzyme units, in accordance with the amount of enzyme that would produce an oxygen uptake of 10 μl per minute when catalyzing the oxidation of 30 μM of ascorbic acid at 25° C. (77° F.) in 3 milliliters of potassium phosphate buffer at pH 5.6. (At 25° C., 1 enzyme unit is equal to 0.405 μM of O_2 per minute.) Therefore, their data showed that 10 μl of oxygen at 25° C. was also equivalent to a change of 3.85 absorbance units at 265 mμ, using ascorbic acid as substrate. The data show that the uptake of oxygen was a linear relationship for the first 10 minutes of the reaction, and that the portions of the curves beyond 10 minutes deviated from linearity as a result of possible substrate depletion.

The relative amounts of ascorbic acid oxidase present in grapefruit and other citrus types indicated that the enzyme concentration varied with fruit size and variety (figure 45). Marsh Seedless grapefruit had the highest concentration of ascorbic acid oxidase, while lemon and lime had the lowest. The concentration of the enzyme on the fresh-weight basis was highest in the smaller young fruit, and decreased with increase in size. The total enzyme concentration per fruit increased during the growth period within a range of 1 to 6 centimeters in diameter. This accumulation of ascorbic acid oxidase in the whole fruit occurred in the flavedo, and disappeared from the albedo and juice.

Glutamic Acid Decarboxylase. Although Axelrod *et al.* (1955) did not report any figures for grapefruit, they did show that the flavedo of lemons and oranges was a rich source of the glutamic acid decarboxylase enzyme. The data in table 75 show that only small differences occurred in the specific activity on a protein nitrogen (PN) basis between navel and Valencia oranges. Lemon flavedo was considerably higher in activity than that reported for either navel or Valencia oranges. Green lemons were more active than ripe fruit. The activity of this enzyme required additional pyridoxal phosphate to prevent loss of activity when it was in contact with its substrate at 37° C. (99° F.) and pH 5.6, but when the reaction was either performed at 15° C. (59° F.) or the pH changed to 6.5, the pyridoxal phosphate was not essential to maintain the initial reaction rate. The glutamic acid decarboxylase enzyme from navel orange flavedo was specific for L-glutamic acid. The optimum pH range at 37° C. was between pH 5.3 and 5.6.

Pectinesterase Activity and Cloud Stability of Grapefruit Juice. Cruess (1914) was one of the earlier investigators to report on the gelation and clarification of citrus juices. He found that fresh orange juice was hard to clarify by filtration until it had stood for a certain length of time (24 to 72 hours). He observed that one sample of orange juice gelled in a few hours after ex-

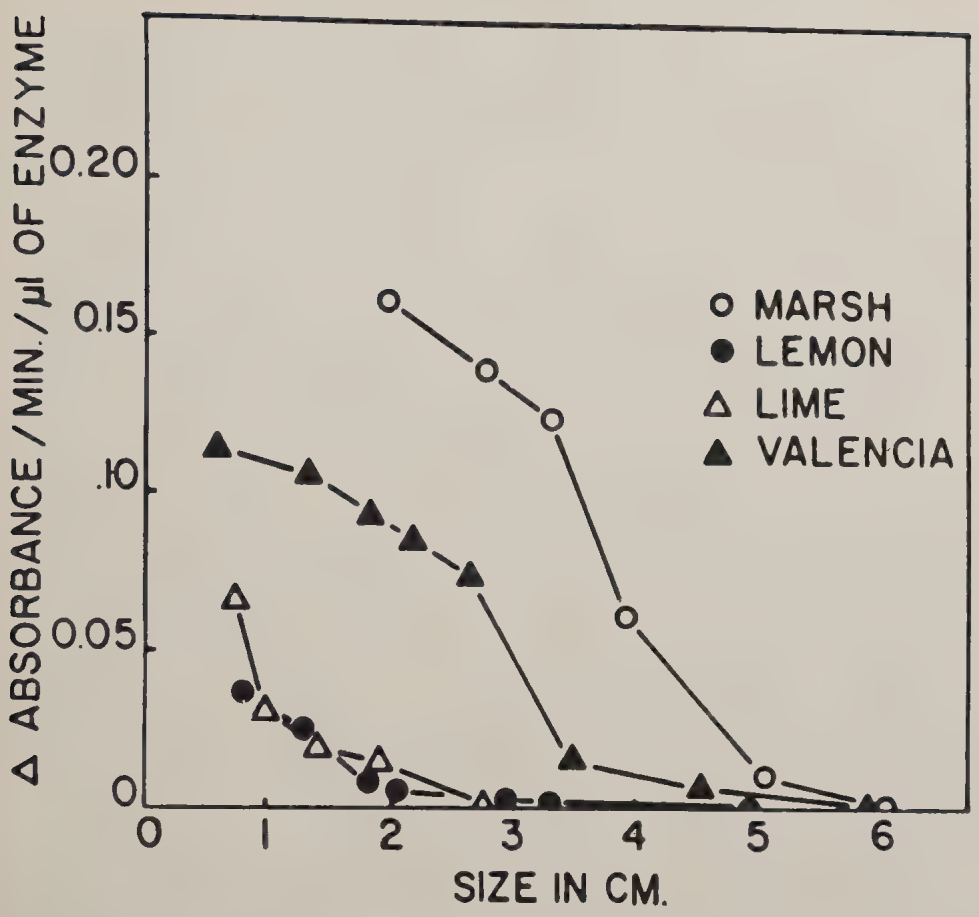


Fig. 45. Ascorbic acid oxidase: A comparison of the enzyme activity among several citrus fruits, ranging in diameter from less than 1 centimeter to 6 centimeters. Enzyme activity is expressed as the rate of change in absorbance per minute at 265 millimicrons per microliter, with ascorbic acid as the substrate. (From Vines and Oberbacher, 1962.)

TABLE 75

SPECIFIC ACTIVITIES OF FLAVEDO GLUTAMIC ACID DECARBOXYLASE FROM VARIOUS CITRUS TYPES.*

Fruits	Milliequivalents $\times 10^{-3}$ grams per minute (fresh weight)	Milliequivalents $\times 10^{-3}$ milligrams protein nitrogen per minute
Navel oranges (fresh).....	5.1	3.7
Valencia oranges (fresh).....	8.4	4.2
Valencia oranges (stored).....	15.3	3.0
Lemons (green).....	10.0	10.0
Lemons (ripe).....	5.5	5.5

*From Axelrod *et al.* (1955).

tracting it from the fruit but, on standing, the suspended material coagulated and settled out, leaving the clear juice. Cruess stated that the enzymes of the juice were the active agents in coagulating and clotting the suspended material. This conclusion was based on the fact that pasteurized juice, in contrast to unpasteurized juice, did not clear on standing because the enzymes were inactivated by heat during treatment.

Processed citrus juices and concentrates that have been reconstituted should retain the cloud of fresh juices and remain free of coagulated pectic substances. The cloud is maintained by inactivating the enzyme pectinesterase, and thus preventing the de-esterification and degradation of the pectic substances. Grapefruit juice contains a sufficient quantity of pectinesterase to demethylate the pectic substances during or after processing unless this enzyme is inactivated, as it may be by heat.

Joslyn and Sedky (1940) published data showing the effects of temperature, pH, and time of heating on the inactivation of enzymes responsible for the clearing of grapefruit juice (table 76). They found that the rate of clearing was slower at higher temperatures or longer heating periods. The heat inactivation of enzymes was more rapid at pH 2.5 than at pH 4.0. Other investigators who studied the control of enzymic flocculation of pectic substances by heating were Parks (1940), Loeffler (1941), Stevens *et al.* (1950), Rouse and Atkins (1952, 1953), and Bissett *et al.* (1953).

It should be noted that the enzyme pectinesterase (also known as pectase or pectin methylesterase) has probably received more intensive study than the other pectic enzymes. This emphasis on pectinesterase activity was prompted by the fact that gelation and clarification of frozen concentrated citrus juices became an important economic problem in establishing the cause of gelation in unpasteurized citrus concentrates. The pectinesterase reacts with the ester linkage to produce methanol and leave a free carboxyl group on the galacturonic acid residue. The esterase activity can be quantitatively determined by titration of the liberated carboxyl groups with a standard alkali. The enzyme activity can also be measured by determining the amount of methanol liberated.

Gelation and clarification changes are highly correlated with the pectic enzymes reacting with the pectin in the juice. From the historical development of this area of research, Joslyn and Pilnik (1961) discussed at some length the causes and prevention of clarification of canned and frozen orange juice concentrates. Only those papers that appear to be pertinent to the enzyme activity of grapefruit juice will be reported in this section.

Rouse (1949) reported that gel formation in frozen grapefruit concentrate which had been thawed and subsequently stored at 40° F. (4.4° C.) was caused by low-methoxyl pectin produced by the action of pectinesterase on the pectic substances in the juice. Figure 46 shows the degree of gelation in frozen

orange and grapefruit concentrate produced under these experimental conditions. The 2 samples that formed gels produced unstable reconstituted juices, which resulted in a water-clear liquid upon separation.

Calcium or other divalent ions are essential for the gelation of low-methoxyl pectin. In low-methoxyl pectin where one-half or more of the ester groups are replaced by carboxyl, the pectin molecule becomes increasingly sensitive to divalent ions such as calcium, and ionic bonding or cross-linking through primary valences becomes possible. The gel structure is influenced by ionic bonding, especially when polyvalent ions are present to unite the adjacent chains. Monovalent cations, such as sodium and potassium, can unite with carboxyl groups, and thus prevent bonding to adjacent molecular

TABLE 76

EFFECTS OF HEATING AT pH 2.5 AND 4.0 ON THE CLEARING OF GRAPEFRUIT JUICE.*

Temperatures of bath		pH	Times of heating to prevent clearing at various temperatures (minutes)	
Degrees C.	Degrees F.		After 6 months	After 12 months
60	140	2.5 4.0	10 10	10 10
65	149	2.5 4.0	10 10	10 10
70	158	2.5 4.0	10 10	10 10
75	167	2.5 4.0	7 10	10 10
80	176	2.5 4.0	2 5	3 6
85	185	2.5 4.0	1 1	1 1
90	194	2.5 4.0	1 1	1 1
95	203	2.5 4.0	1 1	1 1
100	212	2.5 4.0	1 1	1 1

*From Joslyn and Sedky (1940).

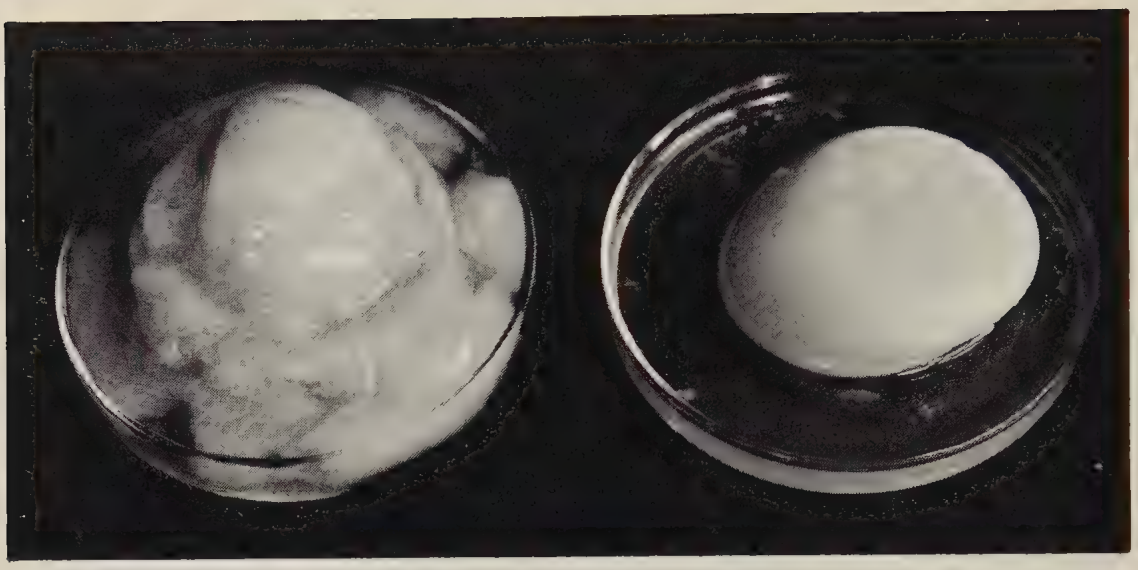


Fig. 46. Low-methoxyl pectin gels in concentrates of Hamlin orange (left, semi-gel) and Duncan grapefruit (right, solid gel) after storage at 40° F. (4.4° C.), resulting from demethylation of pectin by pectinesterase in the presence of a divalent cation (calcium). (From Rouse, 1949.)

chains. The presence of sodium or potassium in the gel system containing calcium interferes with cross-linking, and thus prevents calcium from forming a random gel structure.

The data presented by Rouse (1949) in table 77 indicate that pectinesterase was present and active in unpasteurized frozen grapefruit concentrate. This enzyme will demethylate the pectin molecule to a low-methoxyl product which, in turn, will combine with the calcium in the juice to form calcium pectinate. Under the proper hydrogen ion concentration, this action will produce a gel. The physical and chemical characteristics of the gel depend on the pH and the amounts of low-methoxyl pectin, calcium, and soluble solids present. It should be pointed out that sugar is not essential for the formation of gels with low-methoxyl pectins, but increased amounts of calcium are usually required to produce optimum gels with the reduction of the soluble solids, with the highest calcium requirement in the gels containing no sugar. The effect of added sugar on the optimum calcium requirement of the gels will be to produce pectinates with a methoxyl content between 5.5 and 6.5 per cent (Baker and Goodwin, 1944).

Rouse and Atkins (1952) studied the effects of temperature and time on the inactivation of pectinesterase in Duncan grapefruit juice containing 5 and 10 per cent pulp (table 78). In both types of juice, the enzyme was completely inactivated at 195° F. (91° C.), and at 175° F. (79° C.), even 120 seconds' holding time did not result in complete inactivation. These authors also investigated the effect of pH upon the inactivation of pectinesterase in Duncan grapefruit juice by heat (table 79). Samples of juice with a pulp content

TABLE 77

PHYSICAL AND CHEMICAL PROPERTIES OF FROZEN GRAPEFRUIT CONCENTRATE STORED AT 40° F. (4.4° C.).*

Days stored	0	6	12	18	24	30
SAMPLE 1						
Degree of gelation	None	Slight	Semigel	Solid gel	Solid gel	Solid gel
Separation in 15 minutes (per cent)	40	49	48	50	58	59
Calcium pectate (per cent)	0.16	0.15	0.15	0.10	0.12	0.16
Methoxyl content of pectin (per cent)	4.67	4.46	4.33	3.99	3.41	3.22
SAMPLE 2						
Degree of gelation	None	None	Slight	Slight	Slight	Semigel
Separation in 15 minutes (per cent)	0	28	28	30	40	41
Calcium pectate (per cent)	0.08	0.12	0.08	0.06	0.09	0.12
Methoxyl content of pectin (per cent)	5.96	5.60	4.83	4.28	3.54	2.76
SAMPLE 3						
Degree of gelation	None	None	None	None	Slight	Slight
Separation in 15 minutes (per cent)	0	0	0	2	3	3
Calcium pectate (per cent)	0.10	0.13	0.13	0.14	0.15	0.19
Methoxyl content of pectin (per cent)	7.97	7.20	7.09	7.02	6.87	6.66

*From Rouse (1949).

TABLE 78

HEAT INACTIVATION OF PECTINESTERASE IN DUNCAN GRAPEFRUIT JUICE AT pH 3.5.*

Temperatures		Holding times (seconds)	5 per cent pulp		10 per cent pulp	
Degrees F.	Degrees C.		PEu per degrees Brix (ml × 10^3)	Inactivation (per cent)	PEu per degrees Brix (ml × 10^3)	Inactivation (per cent)
165	74	0	2.7	84.1	5.3	83.4
		5	2.4	85.9	4.6	85.6
		10	2.1	87.7	4.4	86.2
		15	2.0	88.2	4.2	86.8
		30	1.8	89.4	4.1	87.2
		45	1.8	89.4	3.9	87.7
		60	1.6	90.6	3.7	88.4
		90	1.6	90.6	3.5	89.0
		120	1.4	91.8	3.3	89.7
175	79	0	2.4	85.4	4.5	85.9
		5	2.2	86.6	4.2	86.8
		10	2.0	87.8	4.1	87.2
		15	1.8	89.0	3.9	87.8
		30	1.7	89.6	3.5	89.0
		45	1.5	90.9	3.1	90.3
		60	1.3	92.1	2.9	90.9
		90	0.9	94.5	2.6	91.9
		120	0.7	95.7	1.8	94.4
185	85	0	1.8	89.0	3.4	89.0
		3	1.6	90.2	2.8	90.9
		5	1.4	91.5	2.0	93.5
		10	1.1	93.3	1.7	94.5
		15	0.5	97.0	0.9	97.1
		30	0.0	100.0	0.2	99.4
		45	...	....	0.0	100.0
190	88	0	1.1	93.7	2.6	91.6
		3	0.9	94.8	1.6	94.8
		5	0.6	96.6	1.1	96.4
		10	0.0	100.0	0.4	98.7
		15	...	....	0.0	100.0
195	91	0	0.4	97.7	1.1	96.4
		3	0.0	100.0	0.0	100.0
200	93	0	0.0	100.0	0.0	100.0

*From Rouse and Atkins (1952).

of 10 per cent and pH 3.5 were adjusted to pH 3.0 and 3.8, and the activity and percentage of inactivation of pectinesterase in the juice samples were determined at these 3 different pH values. For each temperature studied, the inactivation was decreased as the pH was increased from 3.0 to 3.8.

Pratt and Powers (1953) determined the thermal inactivation curves of pectinesterase and pectic depolymerase of grapefruit juice (figures 47 and 48). These time curves were plotted from the survival–destruction points. The thermal destruction times of pectinesterase of grapefruit juice were 1.06, 1.98, 4.13, and 9.82 minutes at 90, 85, 80, and 75° C., respectively (194, 185, 176, and 167° F.). At the same temperatures, the destruction times of the pectin or pectic acid depolymerizing enzyme of the juice were 1.39, 2.06, 4.23, and 8.49 minutes, respectively.

Published reports seem to indicate that considerable variation exists in the heat stability of pectinesterase in different fruit products.

The relative amounts of pectinesterase activity in the various tissues of citrus fruits are of value to the processor, who needs to know the relationship between enzyme activity and the pectic substances in order to understand the phenomena of gelation and clarification in the production of high-quality citrus concentrates. MacDonnell *et al.* (1945) were the first investigators to isolate and characterize the pectinesterase enzyme of the navel orange, lemon, and grapefruit. They determined the distribution of pectinesterase activities (expressed as pectinesterase units [PEu] per gram of wet weight) in component parts of the grapefruit: flavedo, .049; albedo, .025. The filtered flavedo press liquor and filtered juice fractions contained no pectinesterase activity.

TABLE 79

THE EFFECTS OF pH ON THE HEAT INACTIVATION OF PECTINESTERASE IN DUNCAN GRAPEFRUIT JUICE CONTAINING 10 PER CENT PULP.*

Temperatures		pH 3.0		pH 3.5		pH 3.8	
Degrees F.	Degrees C.	PEu per degrees Brix (ml × 10^3)	Inactivation (per cent)	PEu per degrees Brix (ml × 10^3)	Inactivation (per cent)	PEu per degrees Brix (ml × 10^3)	Inactivation (per cent)
135	57	5.2	86.1	23.9	25.1	33.6	9.9
145	63	4.5	87.9	15.5	51.4	29.0	22.3
155	68	4.3	88.5	7.1	77.7	16.6	55.5
165	74	4.1	89.0	5.3	83.4	6.4	82.9
175	79	3.8	89.8	4.5	85.9	5.7	84.7
185	85	2.8	92.5	3.4	89.0	4.5	87.1
190	88	1.2	96.8	2.6	91.6	4.2	88.7
195	91	0.0	100.0	1.1	96.4	3.3	91.2
200	93	...	....	0.0	100.0	1.5	96.0
205	96	...	....	....	....	0.0	100.0

*From Rouse and Atkins (1952).

This enzyme activity in citrus fruit tissues was found to be associated with the solid particles

Rouse (1953) reported further data on the distribution of pectinesterase activity in various tissues of citrus fruits (table 80). His data showed that, without exception, the juice vesicles had the highest and the centrifuged juice had the least amounts of pectinesterase activity. In the grapefruit va-

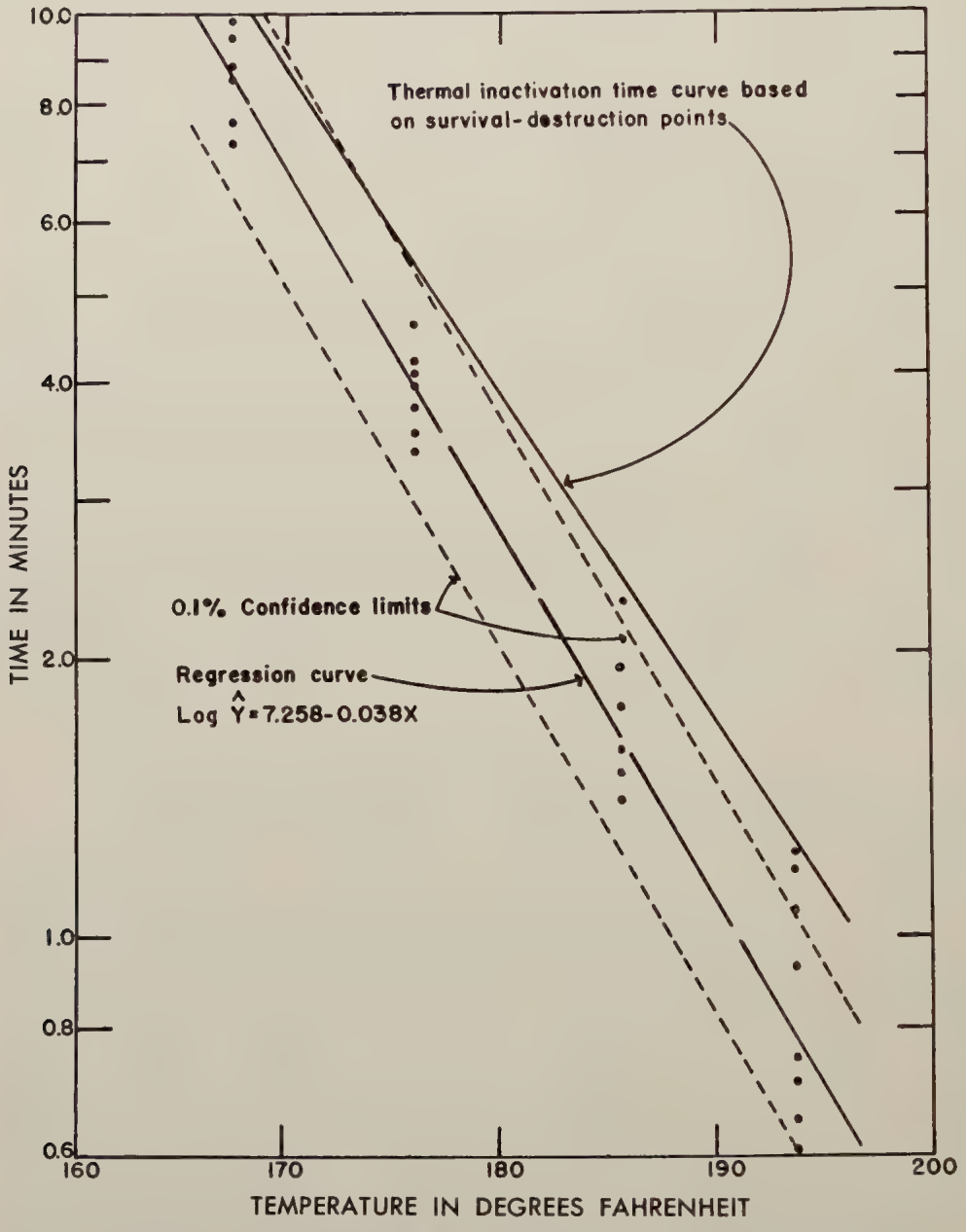


Fig. 47. Thermal inactivation time curve of pectinesterase of grapefruit juice, based on maximum survival points from survival-destruction determinations and regression curve of all survival-destruction points with 0.1 per cent confidence limits. Points shown are the maximum survival points for each batch. (From Pratt and Powers, 1953.)

rieties, the flavedo and albedo exhibited high activity. Consequently, virtually all the pectinesterase activity appears to have been adsorbed on the water-insoluble solids of the tissues. Since pectinesterase activity was associated with the solid particles, the enzyme activity of the juice and concentrate increased with the increase in pulp content, which was composed chiefly of rag and juice vesicles, both of which contained high concentrations of pectin and

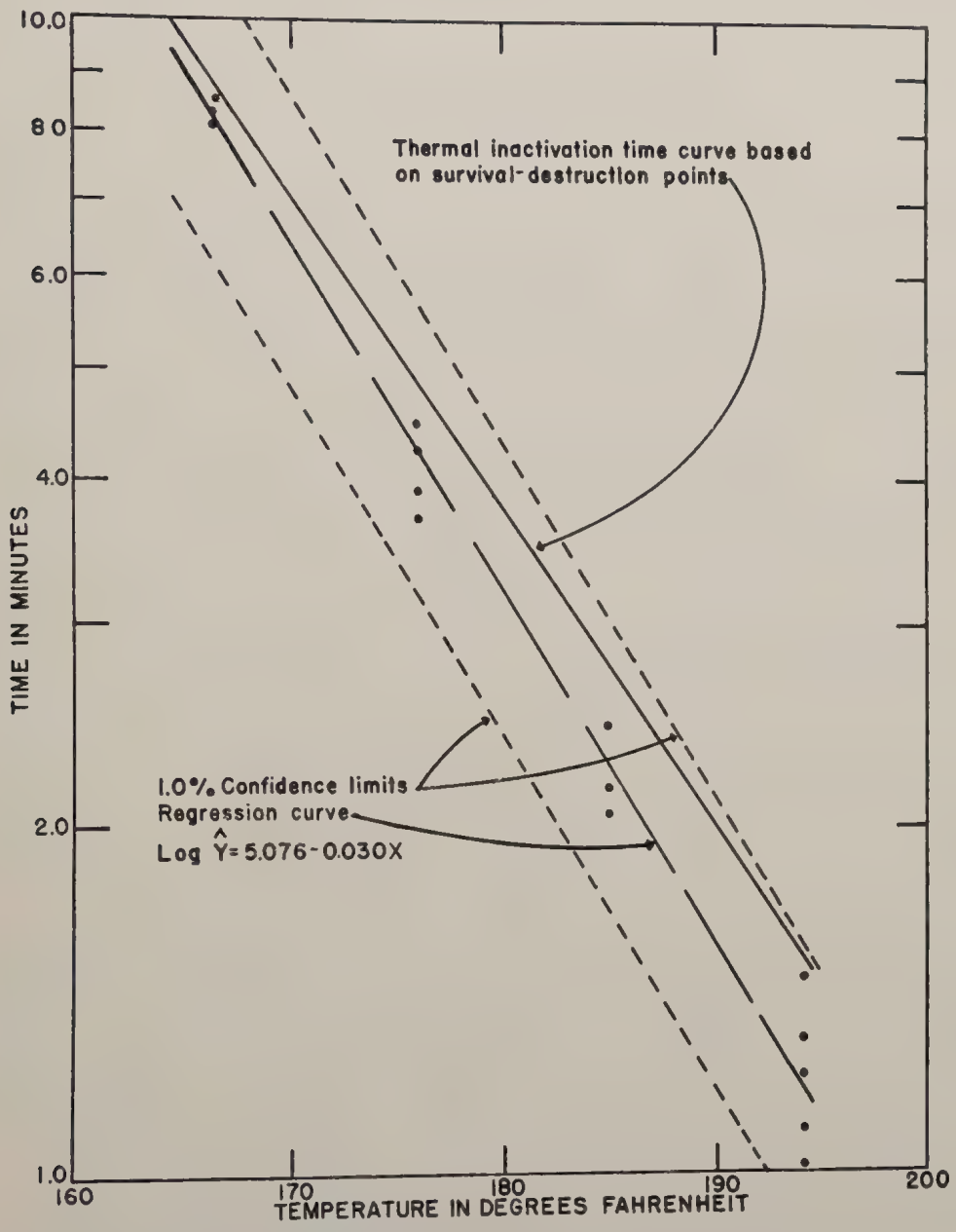


Fig. 48. Thermal inactivation time curve of pectic depolymerase of grapefruit juice, based on maximum survival points from survival-destruction determinations and regression curve of all survival-destruction points with 1.0 per cent confidence limits. Points shown are the maximum survival points for each batch. (From Pratt and Powers, 1953.)

TABLE 80

DISTRIBUTION OF PECTINESTERASE ACTIVITY IN THE COMPONENT PARTS OF CITRUS FRUITS.*

Varieties of fruit	Flavedo		Albedo		Rag		Juice vesicles		Seeds		Juice	
	PEu: g × 10^3		PEu: g × 10^3		PEu: g × 10^3		PEu: g × 10^3		PEu: g × 10^3		PEu: g × 10^3	
	Wet basis	Dry solids	Wet basis	Dry solids	Wet basis	Dry solids	Wet basis	Dry solids	Wet basis	Dry solids	Wet basis	Dry solids
Dancy tangerine	15.8	63.2	16.9	38.7	13.5	66.0	45.7	244.6	4.6	10.3	0.01	0.1
Hamlin orange	15.6	65.8	10.6	43.9	22.8	134.8	55.3	371.9	3.6	6.5	.1	1.0
Pineapple orange	14.6	55.2	8.5	30.4	23.8	109.3	129.9	794.5	6.5	14.2	.4	3.1
Temple orange	10.7	47.4	9.0	34.4	25.6	136.6	75.0	441.4	3.8	7.0	.2	1.6
Valencia orange	9.5	34.4	6.6	20.7	14.1	65.4	76.0	474.3	4.0	8.8	.2	1.9
Marsh Seedless grapefruit	17.2	78.4	12.8	65.6	6.9	39.4	30.0	245.3	0.9	2.2	.2	2.0
Duncan grapefruit	9.8	43.1	11.4	53.1	7.1	41.6	39.9	318.1	2.2	4.7	.3	3.3

*From Rouse (1953).

pectinesterase activity. Based on these experimental observations, the rates of gelation and clarification were thought to be correlated with the increase in pulp content of citrus juices.

As just noted, MacDonnell *et al.* (1945) associated pectinesterase activity with the solid particles in citrus juices. It follows that this activity should increase proportionately with an increase in the pulp content of single-strength citrus juices. This is clearly demonstrated by the curves of figure 49, which show the relationship of pulp to pectinesterase activity in (a) single-strength juice, (b) 42° Brix concentrate, and (c) 42° Brix concentrate stored for 24 hours at 80° F. (27° C.) for 3 different types of citrus juices (Rouse *et al.*, 1954). During the processing of grapefruit juice, the pectinesterase activity was significantly lowered, but no such enzyme reduction was observed in the orange concentrates. Grapefruit concentrates showed a decided decline in pectinesterase activity after 24 hours' storage at 80° F. This decrease was probably owing to the fact that grapefruit concentrate had a higher acidity (pH 3.4) than orange (pH 3.8 and 4.0), thus reflecting the sensitivity of the pectinesterase enzyme to changes in the pH of the juice.

Rouse *et al.* (1965) determined the pectinesterase activity in various tissues of Silver Cluster grapefruit during growth and development for a 10-month period for 2 seasons. Only the data for the juice vesicles are graphically illus-

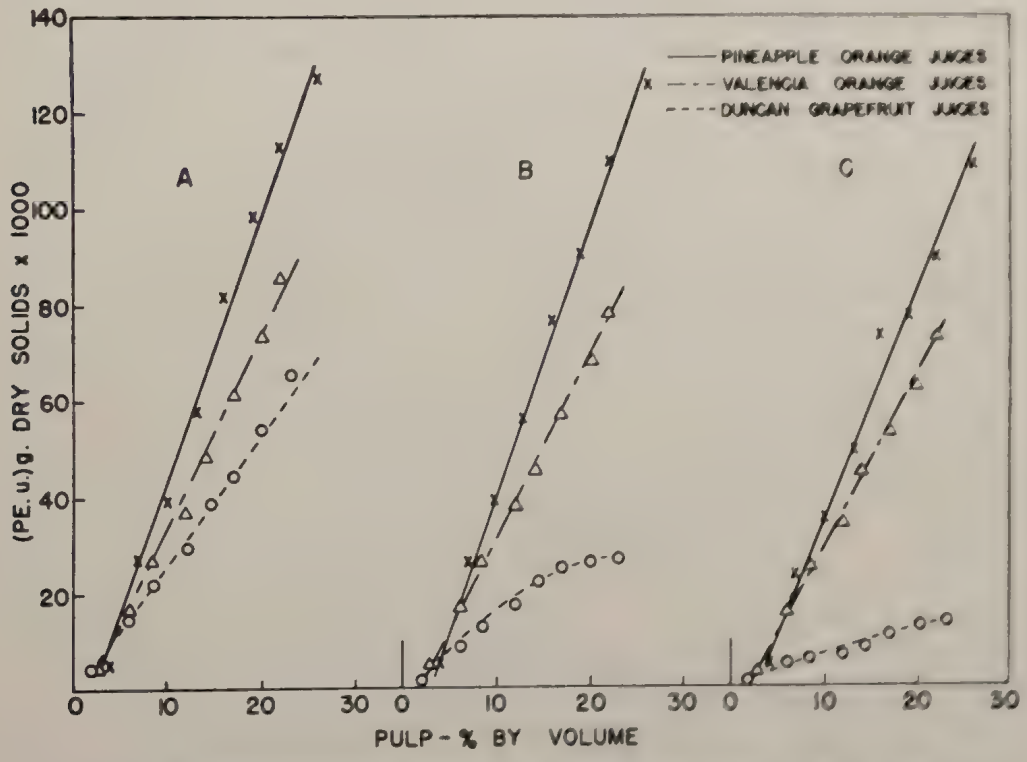


Fig. 49. Relationship of pulp to pectinesterase activity in: *A*, single-strength juices; *B*, 42° Brix concentrates; and *C*, 42° Brix concentrates stored for 24 hours at 80° F. (27° C.). (From Rouse *et al.*, 1954.)

trated in figure 50. Because a large proportion of the pulp in citrus juices is composed of juice sacs, the seasonal changes in the pectinesterase activity of this component are of importance to the processing industry. In the 1961–62 season, the highest pectinesterase activity occurred in August (233 units), followed by a decline for several months and then by an increase in December (195.5 units). The lowest PE activity occurred in March (51.4 units), which value slightly increased for April and May (86.4 units). During this season, the trend in PE activity of the juice sacs continued downward with fruit growth and with the increase in soluble solids:acids ratio. The curve which represents the seasonal changes in activity for the 1962–63 season does not run parallel with that for the 1961–62 season. This difference is the result of a freeze during the 1962–63 season. The pectinesterase activity was 138.2 units in August, 1962, and the values gradually declined to 91.9 units in December, which was the lowest figure recorded before the freeze. Following the subfreezing temperatures that occurred in Florida from December 12 through 14, 1962, the enzyme activity gradually increased to the highest value recorded: 202.5 units in May.

As recorded by Atkins and Rouse (1953), the application of heat to citrus juices to inactivate the pectinesterase enzyme depends upon the temperature obtained and the length of time of exposure. With retention times of 12, 6, and 3 seconds in the pasteurizer, pectinesterase at pH 3.5 in orange juice was completely inactivated at 190 to 205° F. (88 to 96° C.); juices with pH 4.1 required temperatures of 200 to 210° F. (93 to 99° C.). Pectinesterase in Dun-

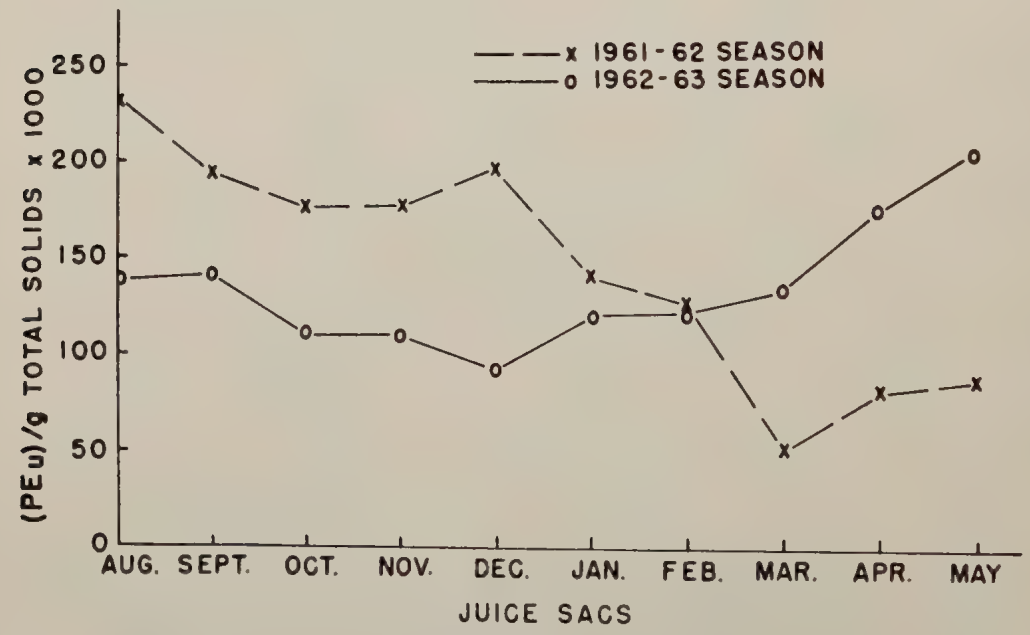


Fig. 50. Changes in the pectinesterase activity of the juice sacs during maturation of Silver Cluster grapefruit in the 1961–62 and 1962–63 seasons. (From Rouse *et al.*, 1965.)

can grapefruit, pH 3.3 and 3.8, was completely inactivated at 190 to 205° F., depending upon the heating time.

In other experiments, these authors expanded their study to include the effect of juice concentration on the thermal requirements for complete inactivation of pectinesterase (Atkins and Rouse, 1954). The data in table 81 show that the percentage of pectinesterase inactivation in seedy grapefruit juice concentrates from 1- to 4-fold became higher with the increase in either the temperature of heat treatment or the retention time in the pasteurizer. The grapefruit juices of 1- to 4-fold showed no indication toward higher enzyme inactivation with decreasing fold or concentration, as had been noted for orange juice concentrates. Pectinesterase in grapefruit juices was completely inactivated at 195 to 205° F. (91 to 96° C.). Note that at 205° F., only 3 seconds' time in the pasteurizer produced complete inactivation.

The soluble-solids content is one of the many variables that affect the cloud stability of citrus juice concentrates. Cotton *et al.* (1947) noted that the total soluble solids affected the rate of clarification of orange concentrate, and that the fastest rate of clarification occurred at 25° Brix. Other investigators (Huggart, 1952; Wenzel *et al.*, 1951) also noted that the cloud stability of orange juice was related to concentration, in that the maximum cloud loss extended to 42° Brix (4-fold) concentration. Grapefruit concentrates were reported to have the quickest rate of clarification within the range of 18 to 41° Brix, and that stability to cloud for 3 months' storage at 40° F. (4.4° C.) was attained only above 60° Brix concentration. Since clarification is one of the changes that may occur in citrus concentrates during processing, transportation, and storage, it is a physical and chemical property that is related to product stability and quality. By measuring the rates of clarification of a series of concentrates of different folds stored under designated conditions, the product with highest stability is determined (Huggart *et al.*, 1951).

The superior cloud stability of orange juice concentrated at levels above 42° Brix (4-fold) has been well established (DuBois and Murdock, 1955; Rice *et al.* 1954). Further interesting data were presented by McColloch *et al.* (1956), in which they studied the relative cloud stability of superconcentrated orange juice (6-fold) and of 4-fold orange juice concentrate. Their data, which confirmed the results of other workers, showed clearly that the cloud stability of heated superconcentrate (6-fold) was exceptionally high, indicating that this product can withstand the adverse conditions which cause excessive cloud loss in the commercial handling of orange concentrate (figure 51). The cloud stabilities of 6-fold unheated superconcentrate were slightly greater than those of the 4-fold heated concentrate. Both these concentrates lost 80 per cent of their initial cloud at 40° F. within 14 days. The unheated 4-fold concentrate lost approximately 85 per cent of its initial cloud in 4 days' storage at 40° F. (4.4° C.).

TABLE 81

TIME-TEMPERATURE RELATIONSHIPS, SHOWING THE PERCENTAGE OF INACTIVATION OF PECTINESTERASE IN SEEDY GRAPEFRUIT JUICE AT VARYING CONCENTRATIONS (pH 3.3).*

Temperatures		1-fold (10.5° Brix) Retention time (seconds)			2-fold (20.5° Brix) Retention time (seconds)			3-fold (29.5° Brix) Retention time (seconds)			4-fold (38° Brix) Retention time (seconds)		
°F.	°C.	12	6	3	12	6	3	12	6	3	12	6	3
125	52	52.0	25.4	18.6	54.2	35.0	23.7	56.1	40.0	37.5	55.2	36.2	25.5
135	57	68.0	63.4	47.1	69.5	65.0	54.2	66.7	59.6	54.2	69.0	63.8	52.9
145	63	74.7	69.0	62.9	72.9	71.5	67.8	70.2	65.4	62.5	74.1	69.0	62.7
155	68	78.7	76.1	75.7	76.3	75.0	73.0	77.2	71.2	66.7	77.6	72.4	64.7
165	74	82.7	78.9	77.1	78.0	76.7	74.6	82.5	78.8	77.1	81.0	75.9	70.6
175	79	89.3	81.7	78.6	86.4	78.3	76.3	91.2	88.5	87.5	93.1	89.7	80.4
185	85	94.7	90.1	84.3	94.9	90.0	86.4	96.5	94.2	91.7	96.5	94.8	90.2
190	88	97.3	95.8	91.4	98.3	93.3	91.5	98.2	96.1	93.7	98.3	96.5	94.1
195	91	100.0	97.2	95.7	100.0	98.3	94.9	100.0	98.1	95.8	100.0	98.3	96.1
200	93	.....	100.0	97.1	.....	100.0	98.3	.....	100.0	97.9	.....	100.0	98.0
205	96	.....	.....	100.0	.....	.....	100.0	.....	.....	100.0	.....	.....	100.0

*From Atkins and Rouse (1954).

McColloch *et al.* (1956) also conducted studies to determine the effect of certain natural juice constituents, such as sugars and acids, on the cloud loss of orange concentrates. The folded orange juice samples were prepared by diluting 58° Brix (6-fold) commercial concentrate with distilled water to known strengths of 12, 23, 33, 42.5, 51, and 58° Brix, which values are represented on the abscissa (*x*-axis) in terms of 1- to 6-fold concentrates (figure 52). The curve labled "orange juice with sugar and citrate added" was constructed from values obtained by adding, to reconstituted single-strength juice, sufficient amounts of a specified sugar-citrate mixture to give a series of juice samples having an effective concentration of 1- to 6-fold. A similar curve was plotted, using data obtained from samples prepared by the same method from a 2-fold orange concentrate.

The 3 series of orange juice samples—the "folded" and the 2 concentrates prepared from 1- and 2-fold juices by adding appropriate amounts of sugars and citrate—were held for 24 hours at 40° F. (4.4° C.). By determining the cloud values before and after the storage period, some interesting relationships were observed between the percentages of cloud loss and fold concentrations of the various samples. McColloch and co-workers confirmed the results of earlier investigators, showing that the rate of cloud loss in orange

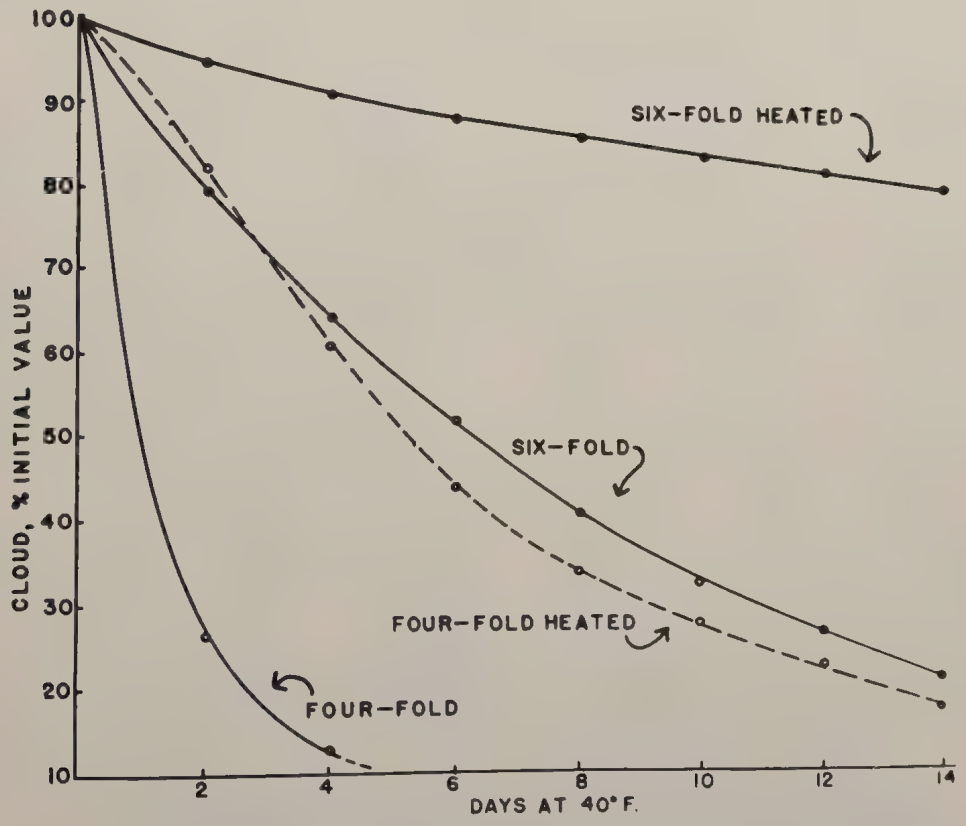


Fig. 51. Cloud changes in orange concentrates at 40° F. (4.4° C.). (From McColloch *et al.*, 1956.)

juices increased from a low rate in single-strength juice to a maximum rate in 4-fold concentrates, and that thereafter, a decreased rate of cloud loss occurred with an increase in concentration. Two of the curves in figure 52 show the relationship between cloud loss and simulated concentrations from 1- to 6-fold in which the total soluble solids of the 1- and 2-fold concentrates were increased to the required amounts by adding a sugar-citrate mixture. The most distinctive characteristic of the 3 curves was that the greatest cloud loss occurred, in each instance, at approximately 4-fold concentration. This observation indicates that the 2 curves constructed from data obtained from

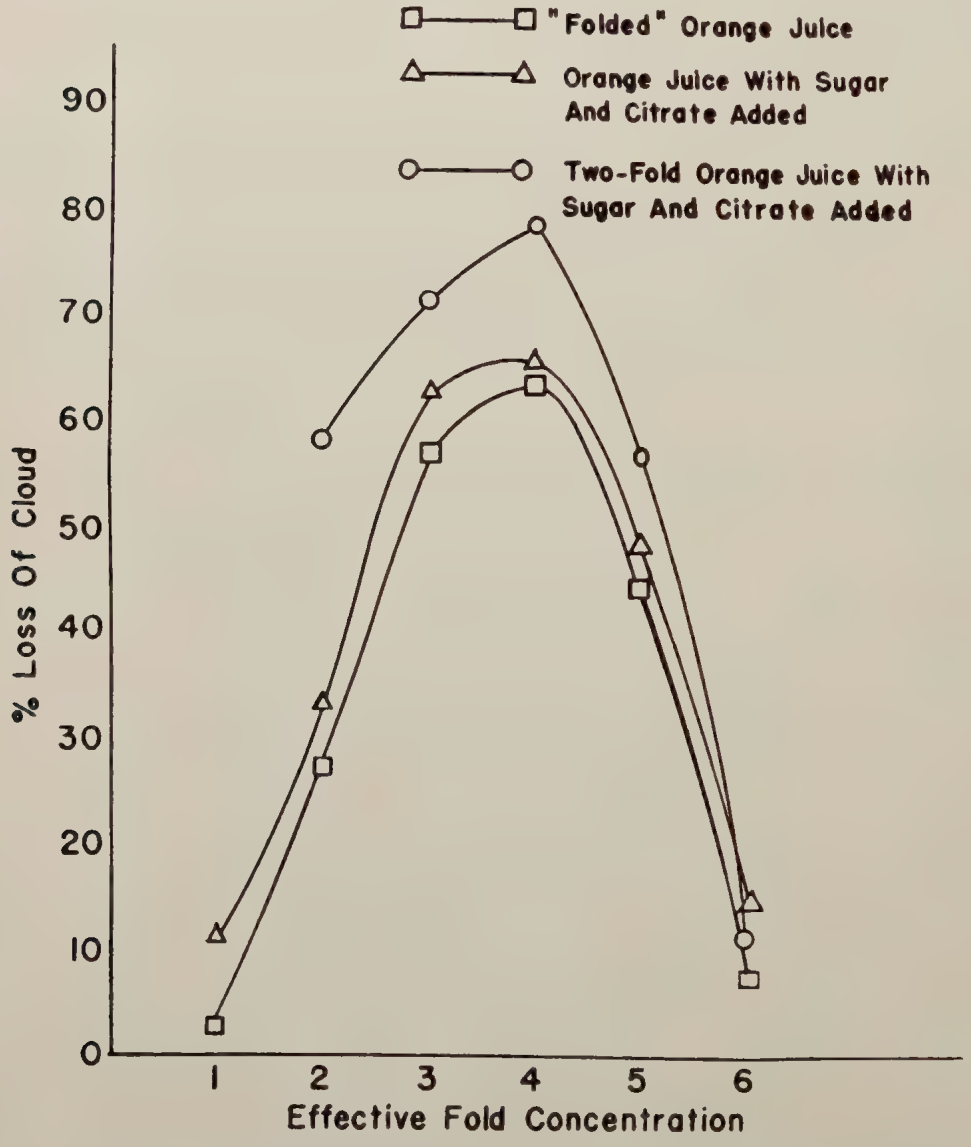


Fig. 52. Comparisons of cloud loss versus concentration in orange-juice concentrates and in concentrates simulated by adding sugars and citrate to orange juices. Twenty-four hours at 40° F. (4.4° C.). (From McColloch *et al.*, 1956.)

folded orange juice, simulated by varying the concentration with sugars and citrate, show the typical cloud-stability curve of folded orange juice.

It appears from the data of these authors that the degree of cloud stability varies with the concentration of juices. Since the concentration of citrus juice products is measured in terms of Brix composed chiefly of sugars and organic acids, it follows that variation in cloud stability is definitely correlated with changing concentrations of sugars and acids (or citrate).

McColloch *et al.* (1956) also studied the effect of calcium ion concentration on cloud stability of 4-fold orange concentrate. Their experiments consisted in adding various amounts of calcium chloride to aliquot portions of 42° Brix concentrate to obtain samples with 0.10 M (control), 0.12 M, 0.16 M, and 0.20 M calcium ions, which were equivalent to 4-, 5-, 6-, and 8-fold concentrations of divalent cation. The samples were placed in storage at 40° F. (4.4° C.), and cloud determinations were made at 0, 17, and 22 hours. Their data, shown in figure 53, indicate that cloud loss was accelerated by increasing the calcium ion concentrations to amounts equal to the divalent cation

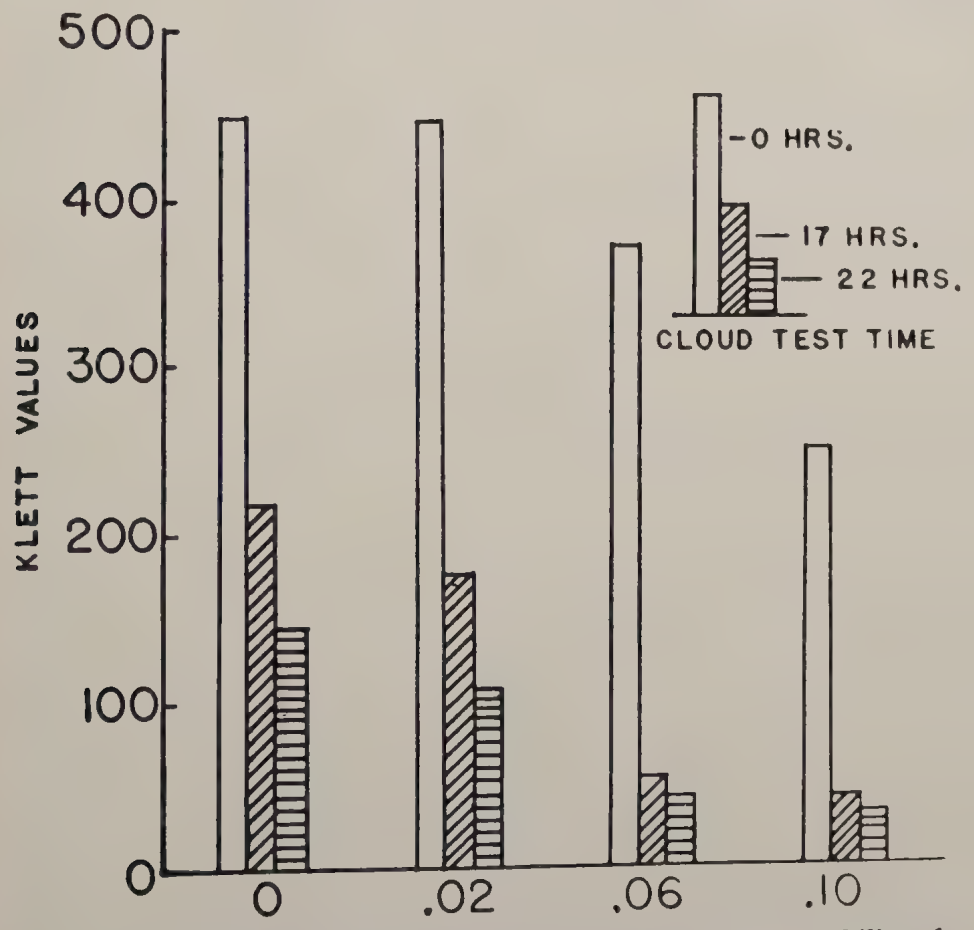


Fig. 53. Effects of added calcium ion on the cloud stability of 4-fold orange concentrate at 40° F. (4.4° C.). (From McColloch *et al.*, 1956.) Bottom figures show moles of calcium ion added.

content of 8-fold concentrate. The data also show that determinations made on samples at zero hours experienced a cloud loss as the calcium ion concentration was increased (see *x*-axis), thus indicating that at sufficiently high concentrations of calcium ions, cloud loss occurred in single-strength juice. They interpreted their results to indicate that the amounts of calcium ions which MacDonnell *et al.* (1945) had found to reduce pectinesterase *in vitro* did not reduce pectinesterase activity in orange concentrate; furthermore, that if the activity was reduced at these calcium ion concentrations, the expected effect on cloud stability did not occur.

Apparently, the rate of clarification and gelation of citrus juice concentrates is definitely related to the pectinesterase activity and pectin content that exist in the product, and these factors become increasingly important, depending upon the conditions of processing and storage of the concentrate. It should also be noted that these 2 factors are partially correlated with the cloud stability of citrus concentrates. By inactivating the pectinesterase enzyme, the soluble pectins of citrus juices remain esterified; conversely, by increasing the pectin content of the juices, cloud stability is increased in the product. The rates of clarification and gelation in frozen citrus concentrates depend upon the concentrations of low-methoxyl pectins in the juices. In low concentrations, the low-methoxyl pectins react with polyvalent cations (metallic ions) to form a flocculent precipitate which occludes the suspended insoluble solids, resulting in the clarification of the reconstituted juice by the complete separation of the solid and liquid phases. At higher concentrations, the low-methoxyl pectins react with polyvalent cations to form a gel structure, which is generally favored by low temperatures. The tendency of this system to form a gel depends upon the affinity of the dispersed phase (water-soluble pectins) and the dispersion medium (the juice serum). If the attraction between the 2 phases is high, the dispersed phase becomes highly solvated and surrounded by shells of solvent, thus increasing the viscosity of the system until a rigid gel structure is formed.

Atkins *et al.* (1953), in their storage experiments with Valencia orange and Duncan grapefruit concentrates containing 5, 10, and 15 per cent pulp, reported that the effectiveness of heat treatments of the juices before concentration prevented gelation and clarification in the concentrates; these effects were found to be dependent upon temperature and the quantity of pulp present in the juices. The degrees of gelation and clarification that occurred in both orange and grapefruit concentrates were primarily dependent upon the amounts of pectinesterase still active in the heated juices. Gelation and clarification were greater in Duncan grapefruit concentrates than in those from Valencia oranges. Pectinesterase traces present in concentrates prepared from heat-treated juices eventually caused marked gelation and clarification when the concentrates were stored at 40° F. (4.4° C.).

The factors just discussed were experimentally investigated by Rouse *et al.* (1957), who showed that cloud stability was positively correlated with the amount of pectin and the intensity of the pectinesterase activity of frozen orange concentrates. A summary of their data is graphically illustrated in figure 54. The histograms show the comparative effects of high and low pectin contents (unshaded and shaded bars) on the cloud stability of orange concentrate (42° Brix) when the comparisons in each pair are made on the basis of equivalent pectinesterase activity. In the 4 pairs of juice samples shown in the graph, the bars are arranged in the order of increasing concentration of the pectinesterase enzyme. All samples with high pectin contents had longer storage periods than those with low contents, even though cloud retention measured in days decreased in both the high- and low-pectin juice samples with increasing pectinesterase activity. Since correlation is a measure of the degree to which these 2 variables vary together, it can be observed that when pectinesterase remains constant in a given experiment, the sample of orange concentrate with a high content of pectin has a longer storage period than the one with low content. Conversely, in 2 samples with the same pectin con-

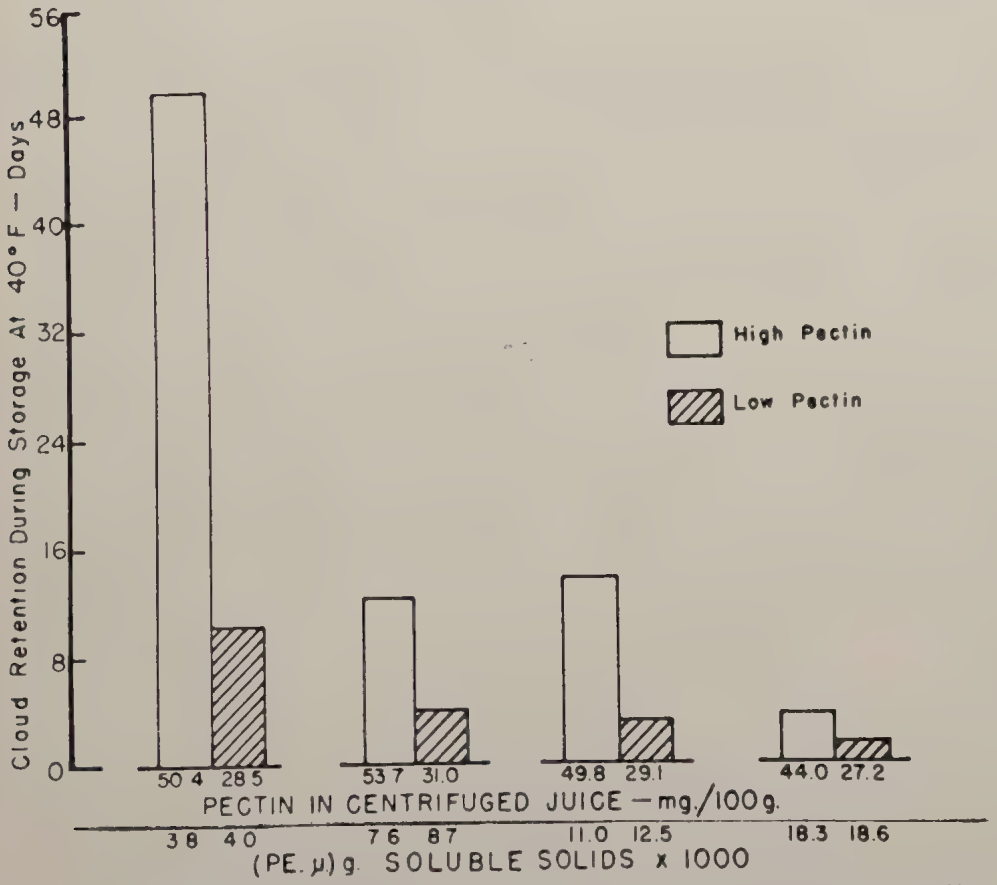


Fig. 54. Retention of cloud during storage at 40° F. (4.4° C.) in 42° Brix commercial orange concentrates with 4 levels of pectinesterase activity and either low or high pectin content. (From Rouse *et al.*, 1957.)

tent, the length of the storage period is related to the pectinesterase activity; i.e., the sample with higher enzyme activity has less cloud stability than the one with low activity (figure 55).

Keller *et al.* (1954) commented on the magnitude of the correlation between pectinesterase activity and cloud stability. Voluminous studies were reported regarding the effects of heat treatment on the inactivation of the pectinesterase enzyme, resulting in increased rates of cloud stability in frozen citrus concentrates. These investigations have led to the general understanding that the pectinesterase enzyme is the cause of cloud loss, and that there should occur a corresponding increase in stability for a given reduction in pectinesterase. It follows that if pectinesterase is the sole cause of cloud loss, a linear relationship should exist between these 2 variables. A highly significant correlation coefficient (+ .893) was calculated from their data. In spite of this high correlation, they noted that it was difficult to predict any particular cloud-stability value on the basis of the absolute pectinesterase activity in processed products.

These variables are further discussed in the following quotation taken from their paper.

It is very probable that PE is involved in the cloud-loss process, but it is also possible that other independent factors are involved in the *prevention* of cloud loss by heat treatment other than the inactivation of PE. Therefore, a correlation between

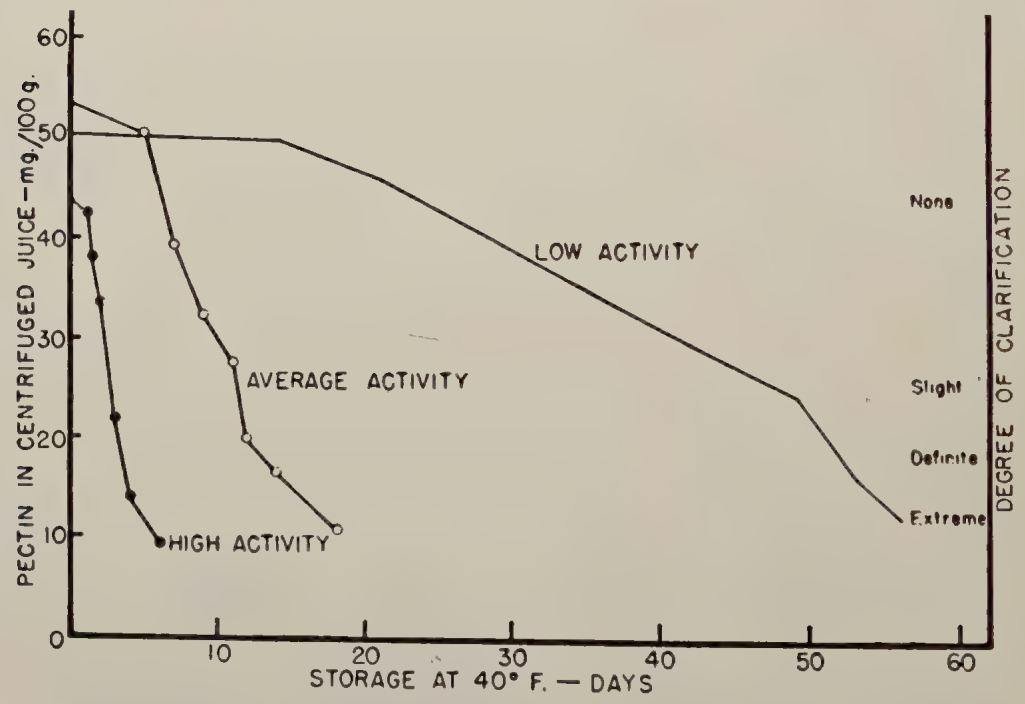


Fig. 55. Effects of high pectin content on the storage life at 40° F. (4.4° C.) of 42° Brix commercial orange concentrates containing 3 levels of pectinesterase activity. (From Rouse *et al.*, 1957.)

the amount of PE inactivated and cloud stability does not indicate how important PE is to the cloud-loss mechanism, or that PE alone causes cloud loss, or whether PE in combination with other factors, such as heating, homogenization, etc., brings about this relationship. It is known only that PE acts upon pectin to bring about changes which could result in cloud loss, but the observation that a correlation exists between the amount of PE remaining after heating with increased cloud stability does not evaluate the role of the causes involved. It simply determines the regularity with which the amount of PE was in accord with the cloud-loss rate, based on the supposition that all the factors involved in the cloud mechanism could be lumped together as cloud-loss rate vs. PE.

Carotenoids as Pulp Pigments

CITRUS FRUITS owe their color to the pigments which are located in various portions of the fruit, but the coloring matters are usually found in the cells of the outer peel (flavedo) and in the juice vesicles. The pigments are unequally distributed throughout the cells, but are concentrated in the plastids and chromatophores. The physical properties of the plastids appear to be species characteristics. Consequently, as shown in the following text, not all the coloring matter of citrus fruit is in the plastids. In some instances, the pigments are in the cell sap and cell walls.

The carotenoids are hydrocarbons containing 40 carbon atoms, such as carotene (named after carrots) and its isomer lycopene (the red pigment of tomatoes) and their alcohol, ketone, and ester derivatives. Their color varies from pale yellow to deep red. All other so-called carotenoids are probably decomposition products with fewer than 40 carbon atoms.

Carotenoids are usually insoluble in water, but are soluble in organic solvents such as acetone and ether. Most plant carotenoids are found in the plastids and not in the cell fluids. They are usually present in colloidal suspension in the cell lipoids, or are mixed with solid or semisolid fats. Their chemical structure is composed alternately of single and double bonds between the carbons in the chain constituting the molecular skeleton. The degree of color depends upon the number of double bonds in the conjugated system and the concentration of carotenoids that occurs in the tissues under experimentation. A minimum of 7 bonds must be conjugated before a perceptible color appears; consequently, related polyenes with fewer bonds in the conjugated state are technically not carotenoids.

The carotenoids are, therefore, composed of tetraterpenes containing 40 carbon atoms in which the isoprenoid (methylbutadiene) pattern is discernible in the molecule, and this pattern is repeated 8 times in the chemical structure of the C_{40} carotenoids. The terminal carbon atoms on each end of the carotenoid chain may be closed to form ring systems, as in β-carotene, or they may also exist in an open chain form rather than in a ring structure, as in lycopene, the red pigment of the tomato. The 40-carbon isoprenoid chain

has a central point of symmetry which is common to all carotenoids. There are 2 classes of carotenoids: (1) the carotenes which are hydrocarbons of the $C_{40}H_{56}$ type and of the C_{40} type which are more highly hydrogenated; and (2) the xanthophylls, which are oxygen derivatives of the carotenes.

The structural relationship of the 4 natural isomers of $C_{40}H_{56}$ that have been isolated can be observed in figure 56. Lycopene and β-carotene are both $C_{40}H_{56}$ compounds, but the basic chemical difference between the 2 compounds is that the former is aliphatic in character, and can be hydrogenated to a paraffin $C_{40}H_{82}$, while the latter is alicyclic and, when fully hydrogenated, yields $C_{40}H_{78}$. Accordingly, lycopene has 13 double bonds and carotene has 11, and this difference is owing to the 2 terminal ring structures, 1 at each end of the chain in β-carotene. Further, γ-carotene is different from lycopene in that the former has 1 β-ring configuration (β-ionone group) introduced by

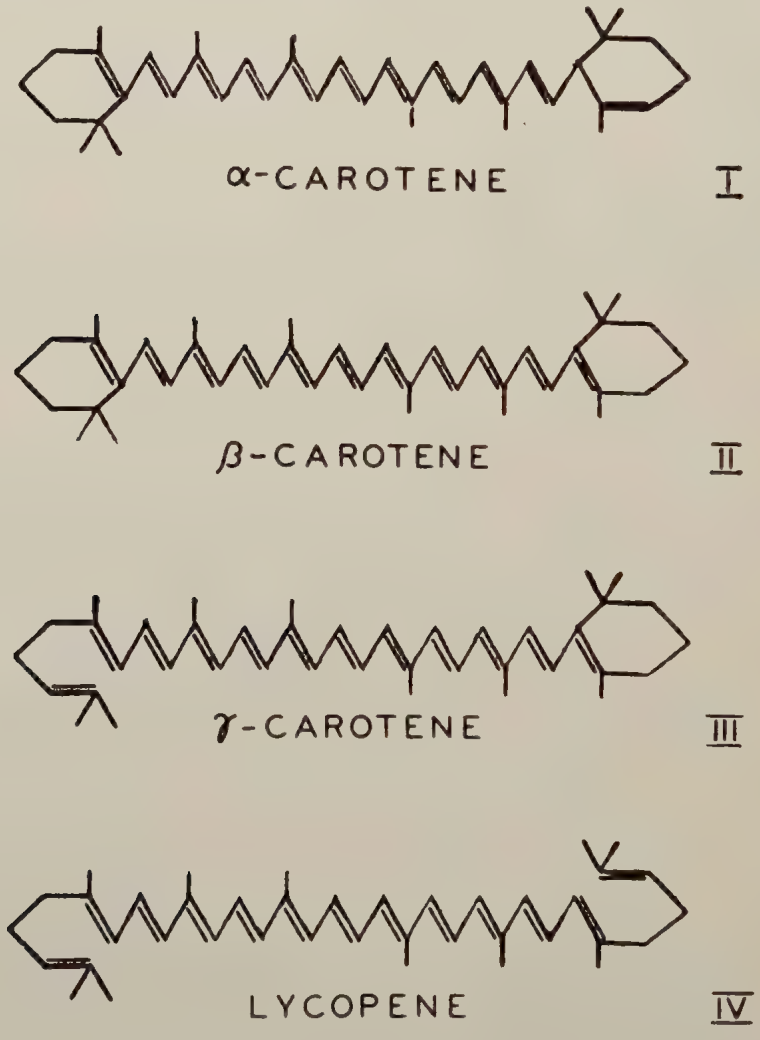


Fig. 56. The structures of some natural isomers of $C_{40}H_{56}$ hydrocarbons that have been isolated. (From Mackinney, 1961.)

ring closure at 1 end of the long chain. In the α-carotene molecule, 1 end of the long chain is attached to an α-ionone ring, and the other end is attached to a β-ionone group. (See Mackinney, 1961.)

Carotenoids are discussed here as pigments of the pulp. Carotene as provitamin A in edible portions of the grapefruit is briefly noted on page 114 of this chapter, and in the seed and peel starting on page 221 of chapter 3. For more detailed information, see Karrer and Jucker (1950).

The pink and red grapefruit pigments are of interest to juice processors. They appear to be stable in the frozen concentrate, but not in the canned, single-strength juice. In the latter, they may give the juice a muddy color, and it often turns brown after a few months in storage.

Traub *et al.* (1929) reported that the pink pigment in Foster and Thompson (Marsh Pink) was apparently a carotenoid. Matlack (1935) isolated and determined the pigments in the same 2 varieties; his spectrographic analysis showed that both contained lycopene and β-carotene. Lycopene was most abundant in Foster and carotene in Thompson. This explains why Foster is true pink and Thompson is salmon-pink. Lycopene is confined chiefly to the segment membranes, and often may be seen through the peel. Matlack identified the two pigments by making spectrographic comparisons of lycopene from grapefruit and tomato, and of carotene from grapefruit and carrot. They agreed very closely, as may be seen by the values in table 82.

Marie Jones (1951) reported that half a teaspoonful of red crystals, which proved to be lycopene, had been isolated from red grapefruit (variety unspecified) by workers in the United States Fruit and Vegetable Products Laboratory at Weslaco, Texas.

Swift (1946) obtained 50 per cent more carotenoids from grapefruit pulp by using a burr reamer than by using a Faulds rotary press.

TABLE 82

COMPARISONS OF ABSORPTION MAXIMA OF LYCOPENE FROM TOMATO AND GRAPEFRUIT AND OF β-CAROTENE FROM CARROT AND GRAPEFRUIT.*
(Data are stated in angstrom units.)

Band	Lycopene			β-Carotene		
	Tomato	Grapefruit		Carrot	Grapefruit	
		Foster	Marsh		Foster	Marsh
I	5,205	5,210	5,205	5,000	5,000	4,998
II	5,020	5,025	5,025	4,670	4,668	4,670
III	4,845	4,840	4,830	4,380	4,380	4,380
IV	4,545	4,550	4,550	4,130	4,130	.....
V	4,280	4,283	.....	.....	.....	.....

*From Matlack (1935).

Khan and Mackinney (1953) determined the carotenoids and the proportions in which they were found in 4 varieties of grapefruit (table 83). They extracted the flesh with acetone. The carotenoids were transferred to petroleum ether and subsequently chromatographed on magnesium oxide Hyflo Super Cel columns. The individual components that were separated were calculated from the optical densities at the respective maxima by the use of specific absorption coefficients.

The absorption curves of the petroleum ether extracts showed the existence of significant differences in the amounts of individual carotenoids in the pulp of different grapefruit varieties. The absorption curves of the Ruby variety showed characteristic bands with maxima *ca.* 503, 472, and 448 mμ. The shapes of these curves were similar to those produced by a 1:1 mixture of lycopene and β-carotene. The crude extract of Coachella Pink (Redblush) yielded a 3-banded spectrum at 478, 450, and 425 mμ, which indicated the predominance of β-carotene. A crude extract of Marsh White grapefruit showed a spectrum of 3 well-defined bands at 425, 400, and 380 mμ.

The pigment contents of the 4 grapefruit varieties are shown in table 83. The 3 pink varieties were noteworthy in that they contained large amounts of β-carotene and lycopene, and relatively small amounts of ζ-carotene and phytofluene. Traces of α- and γ-carotenes and other components were observed. Lycopene and β-carotene were not present in the Marsh White variety. They found a pale-yellow component in the white strain with an absorption close to that of lycopene, but ζ-carotene and phytofluene were the pigments determined.

Lime *et al.* (1954) investigated the development and fading color in Ruby (Ruby Red) grapefruit in relation to changes in composition of the edible portions of the fruit during growth and maturity. They showed that optimum qualities of both color and flavor of canned juices were related to seasonal changes in composition of the fruit (table 84). Figure 57 shows the index of fading in relation to the lycopene and carotene contents of the Ruby variety during growth and maturity in the 1953–54 season. The amount of lycopene

TABLE 83

PIGMENT CONTENT OF GRAPEFRUIT PULP.*
(In micrograms per 100 grams.)

Varieties	Lycopene	β-Carotene	ζ-Carotene	Phytofluene
Texas Pink†	237	207	18	5
Redblush	176	142	10	20
Coachella Pink	28	129	14	3
Coachella White	...	tr	6	9

*From Khan and Mackinney (1953).
†Texas Pink is a trade name; Coachella Pink is Marsh Pink; Coachella White is Marsh White.

TABLE 84

CHANGES IN COMPOSITION OF THE EDIBLE PORTION OF RUBY RED GRAPEFRUIT DURING GROWTH AND MATURITY.*

Dates of samples	Average weight (grams)	Acid (per cent)	Degrees Brix	Brix:acid ratios	Naringin (per cent)	Lycopene (mg/100 grams)	Carotene (mg/100 grams)
1953:							
July 27.....	207	2.43	12.5	5.1:1	.....	0.93	0.07
August 11.....	215	2.09	12.2	5.8:1	.....	1.00	.08
August 24.....	266	1.93	11.6	6.0:1	0.075	1.15	.10
September 8...	298	1.68	10.4	6.2:1	.068	0.91	.12
September 21..	337	1.63	10.3	6.3:1	.058	.91	.14
October 5.....	370	1.49	10.3	6.9:1	.051	.72	.14
October 19.....	357	1.39	10.1	7.2:1	.047	.38	.23
November 2...	407	1.37	10.1	7.4:1	.043	.49	.31
November 16..	429	1.33	10.0	7.5:1	.041	.17	.31
November 30..	461	1.32	9.8	7.4:1	.039	.16	.34
December 14...	479	1.25	9.8	7.8:1	.034	.21	.31
December 28...	484	1.31	9.6	7.3:1	.038	.19	.37
1954:							
January 11.....	477	1.23	10.2	8.3:1	.038	.19	.34
January 25.....	515	1.29	10.2	7.9:1	.033	.11	.33
February 8	543	1.23	10.2	8.3:1	.033	.03	.33
February 23...	535	1.27	10.4	8.2:1	.029	.10	.31
March 8.....	538	1.13	10.4	9.2:1	.027	.10	.34
March 22.....	534	1.02	10.2	10.0:1	.023	.05	.23
April 5.....	517	1.05	10.0	9.5:1	.028	.08	.22

*From Lime *et al.* (1954).

increased rapidly during August, and subsequently decreased sharply until the middle of November, with only a slight decrease from November to April. The carotene content gradually increased from July through January, and remained nearly constant until the middle of March before the curve declined. The lycopene and carotene curves intersected early in November and, from then on, the concentration of carotene exceeded that of lycopene in the samples. Maximum color occurred in Ruby from the latter part of September through October, a period in which the fruit was high in naringin and immature according to the Brix:acid ratio. The pulp color began to fade in November, and continued to fade as the season advanced.

Purcell and Schultz (1964) determined the seasonal changes in the lycopene content of off-bloom and normal Texas red-fleshed grapefruit, and concluded that neither the season nor the actual fruit age was the sole controlling factor in initiating the decline of lycopene concentration, but that both factors exerted an influence.

In studying the effect of artificial climate on the internal color of Redblush grapefruit, Purcell *et al.* (1968) designed an experiment to determine the effect of temperature on the lycopene content of the fruit during growth and development. The experimental material used in this study consisted of small potted trees with grapefruit grafted onto them. These were placed in climatic chambers which were regulated at various day and night temperatures. During the growth period, fruit samples were taken from each chamber at 2-week intervals for the determination of lycopene. The results of these experiments are graphically summarized in figure 58, which shows the comparative lycopene concentrations in fruits from plants in the controlled environmental chambers. The lycopene content at a day temperature of 95° F. (35° C.) and night temperature of 85° F. (29.4° C.) increased immediately and significantly, and reached a maximum at the beginning of November (near the end of the experiment). Conversely, the lycopene content of fruit at 70° day and 60° F. night temperatures (21 and 16° C.) decreased immediately and continuously during the sampling period, approaching zero value during September. The reaction of fruit grafted onto seedlings in the 60/40° F. chamber (16/4.4° C.) was similar to the reaction of fruit borne on trees in

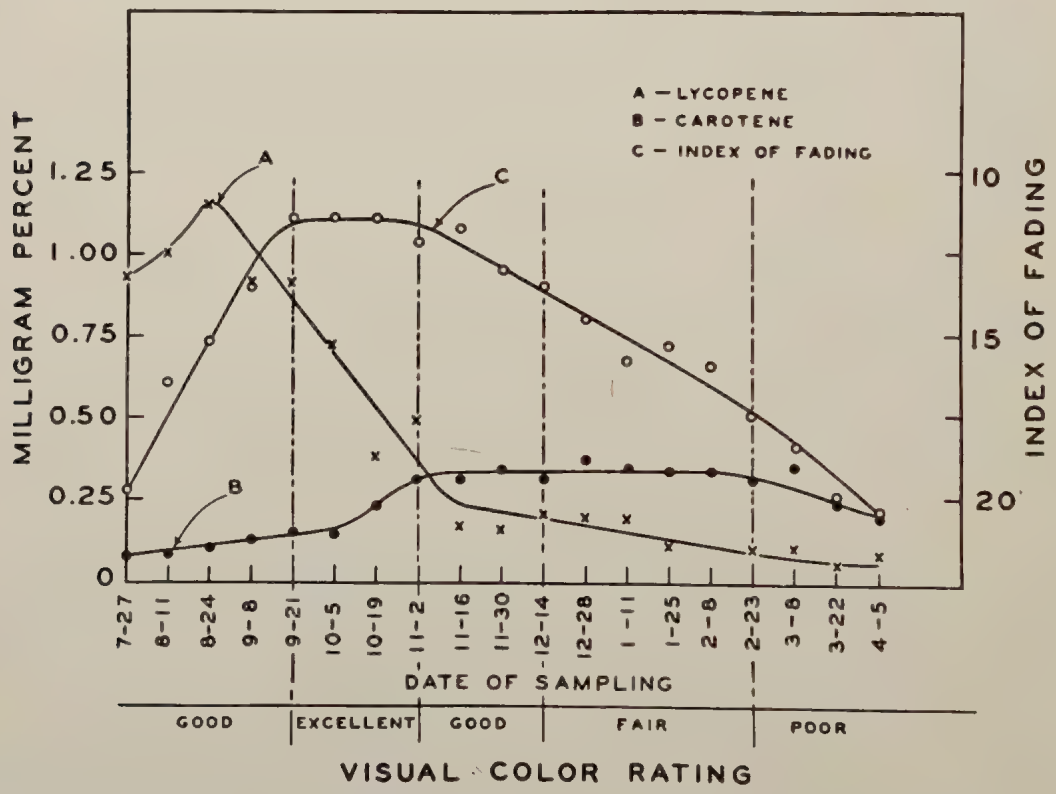


Fig. 57. The interrelationships of index of fading, visual color, and lycopene and carotene content of Ruby Red grapefruit during growth and maturation in the 1953–54 season. (From Lime *et al.*, 1954.)

the preceding season. The lycopene content per fruit from both the grove and from the grafted, controlled plants (held outside in full sunlight) reached maximum values at about the middle of October before declining. The linear and quadratic trends in the time-course study of lycopene production in both grove and grafted, controlled fruit were shown to be statistically different, but both curves followed the same pattern of seasonal production.

These authors postulated 2 metabolic systems to explain the synthesis and destruction of lycopene during grapefruit maturation. They suggested that

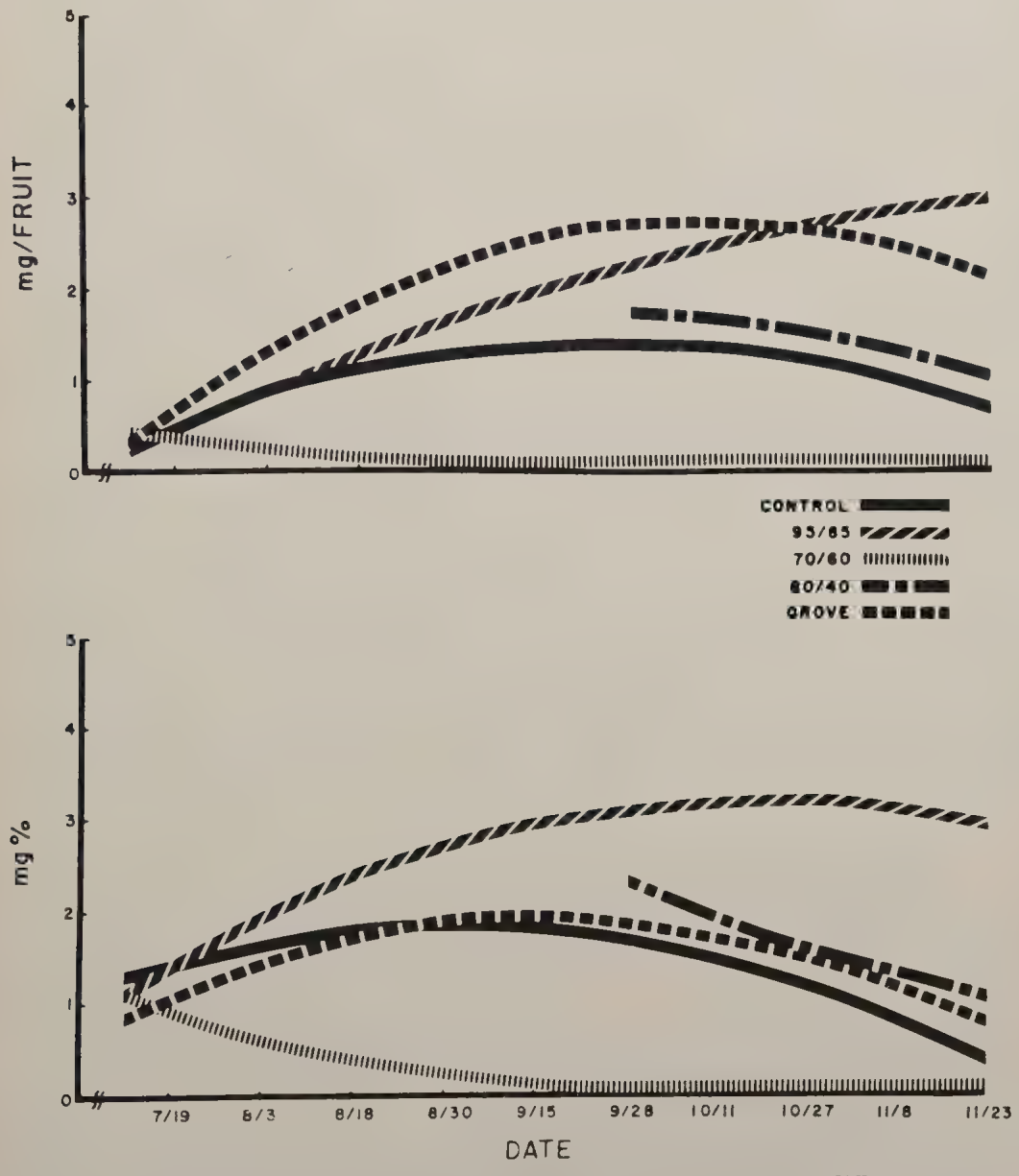


Fig. 58. *Top:* Lycopene yield of Redblush grapefruit under different temperatures, plotted against dates. *Bottom:* Lycopene concentration in grapefruit carpels under different temperatures, plotted against dates. (See text for temperature details.) (From Purcell *et al.*, 1968.)

both processes probably occurred simultaneously during certain intervals of the maturation period. The results reported in figure 58 indicate that the lycopene-destroying system was initiated at a certain stage of fruit development, and that the rate of reaction gradually increased with age. The maximum lycopene yield occurred at the point where the 2 rates, that of synthesis and that of destruction, were equal. Higher temperatures might either increase the rate of lycopene synthesis or reduce the rate of reaction of the lycopene-destroying system. Periods of low temperature would have the opposite effects on these reactions.

The rates of seasonal change in the carotenoid pigments of grapefruit are similar to those in the orange. Miller *et al.* (1941) reported that the carotenoid pigments in the juice of Florida oranges gradually increased during the season up to the time when the juice was palatable, which was about March. When the highest concentrations of carotenoids were attained, the amounts present varied from 3 to 4 times as much as in the September samples.

It can be observed that the concentrations of pigments present in citrus fruits vary, in common with other seasonal components, with variety, rootstock, stage of maturity, and environment, but it is certain that the total variability that has been noted within this system has not been equally divided among these factors.

Curl and Bailey (1957) fractionated the carotenoid pigments of Ruby Red grapefruit pulp and peel by countercurrent distribution in a glass Craig apparatus, a method which produced a preliminary separation of the carotenoids into several fractions, but chromatography was subsequently used to make the final separation and purification of the constituents. The fractionation results are reported in table 85. The hydrocarbons were the principal components of the carotenoid pigments in both the pulp and peel, but relatively small quantities of the monol and diol (plus polyol) fractions were

TABLE 85

COUNTERCURRENT DISTRIBUTION OF SAPONIFIED CAROTENOIDS FROM RUBY RED GRAPEFRUIT PULP AND PEEL.*

Carotenoids	Approximate percentages of total carotenoids (measured as β-carotene)	
	Pulp	Peel
Hydrocarbons (I)†.....	93	76
Monols (II-A).....	3	7
Monoether monols (II-B)...	..	3
Diols and polyols (III).....	4	14

*From Curl and Bailey (1957).
†Roman numerals in parentheses denote fractions, as recorded by the authors.

TABLE 86

CAROTENOIDS OBTAINED FROM RUBY RED GRAPEFRUIT PULP AND PEEL.*

Fractions	Constituents	Approximate percentages of total carotenoids	
		Pulp	Peel
I-1	Phytoene	16.0	47.0
I-2	Phytofluene	4.4	14.0
I-3	α-Carotene	....	0.1
I-4	β-Carotene	27.0	7.2
I-5	Phytofluene-like	....	.4
I-6	ζ-Carotene	3.5	7.2
I-7	γ-Carotene-like	0.8	.4
I-8	Lycopene	40.0	11.0
II-1	Substance 377	.2	....
II-2	Substance 335	.6	.5
II-3	"Hydroxy-α-carotene"	.2	.1
II-4	Cryptoxanthin	.7	1.4
II-5	Cryptoxanthin-like	.4	....
II-6	Cryptoflavin-like	....	1.3
II-7	Rubixanthin-like	.5	.3
II-8	Cryptochrome-like	....	.2
III-1	Lutein	.3	.9
III-2	Antheraxanthin	.7	....
III-3	Violaxanthin (+ zeaxanthin?)	.9	1.0
III-4	Luteoxanthins	.4	1.8
III-5	Mutatoxanthins	.4	.2
III-6	Valenciachromes	.2	.3
III-7	Trollichrome-like	....	.2
III-8	Auroxanthins	.3	1.6

*From Curl and Bailey (1957).

noted. Higher percentages of both monols and diols (plus polyols) were obtained from the peel carotenoids. As with their results on orange peel (Curl and Bailey, 1956), these researchers found a monoether monol fraction in grapefruit peel.

The carotenoids in Ruby Red grapefruit were obtained by chromatographing the 3 pulp and 4 peel fractions that had already been determined by countercurrent distribution (table 86). The values reported for the pulp carotenoids confirmed data, published earlier, that the 2 principal pigments in pulp tissue were lycopene and β-carotene, with significant amounts of phytofluene and ζ-carotene, as noted by Khan and Mackinney (1953), cited in connection with table 83. Phytoene accounted for 16 per cent of the total carotenoids of the pulp and 47 per cent in the peel, followed by phytofluene

with 4.4 and 14 per cent, respectively. Identified in the peel only was α-carotene, and a small quantity of material was obtained from both pulp and peel which appeared to be γ-carotene. Many minor constituents were obtained in low concentrations from both pulp and peel.

Factors Influencing the Internal Color of Red and Pink Grapefruit. Reference has been made in this section to the data of Lime *et al.* (1954), who reported on the seasonal decrease of the internal (flesh) color of Texas Ruby Red grapefruit; and to the data of Matlack (1935), who attributed the red and pink colors of pulp predominantly to lycopene and β-carotene. (See tables 82, 84, and figure 57, with related text.) It is well established that the seasonal changes of grapefruit are significantly demonstrated by changes in the physi-

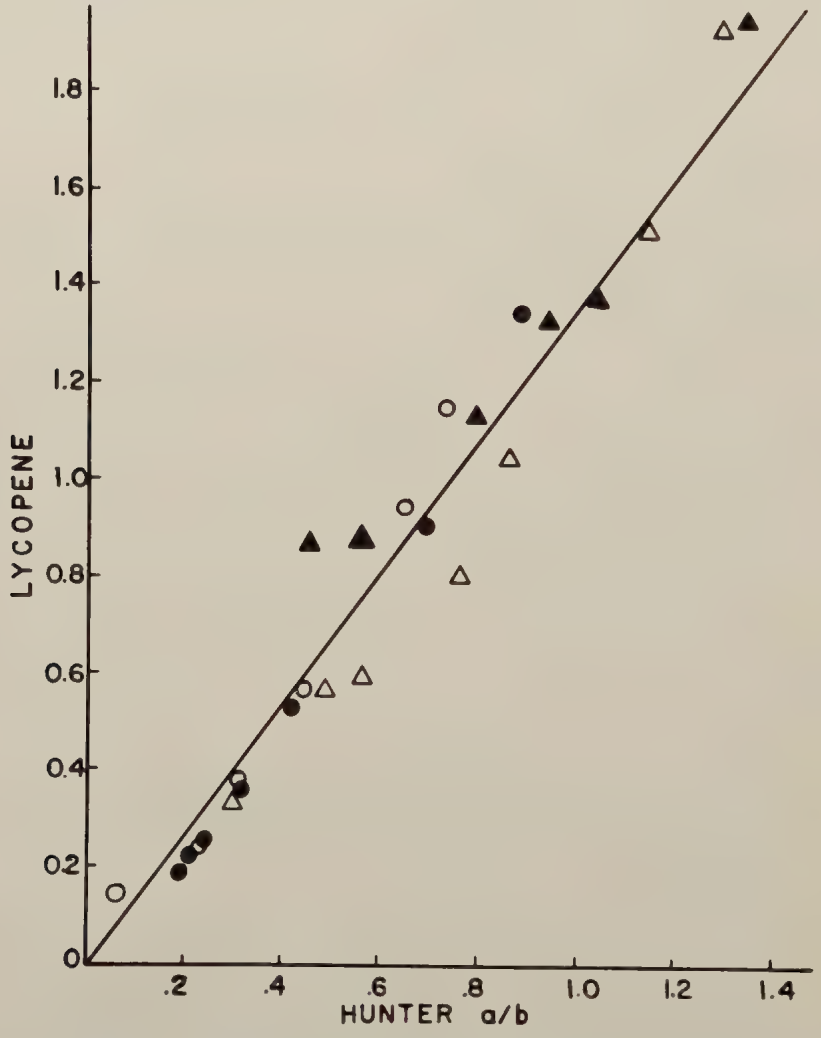


Fig. 59. Relationships between Hunter a/b ratios and lycopene content of red and pink grapefruit on 2 different rootstocks. Black triangles indicate red on rough lemon; open triangles, red on sour orange; black circles, pink on rough lemon; and open circles, pink on sour orange. (From Ting and Deszyck, 1958.)

cal, chemical, and compositional factors of the fruit. Hence, such changes in the internal color and pigment content of red and pink grapefruit should also be influenced, to various degrees, by variety, rootstock, size of fruit, age of trees, maturity, and locality.

The foregoing factors were correlated with the internal color development of Florida red and pink grapefruit by Ting *et al.* (1958). They measured the internal color of the fruit with the Hunter color-difference meter, a tristimulus photoelectric colorimeter. The same method was used earlier by Huggart and Wenzel (1955) to measure color differences in various citrus juices and concentrates. The 3 values used in the Hunter method to express the results are: the R_d expression, which is the diffuse reflectance or brightness value; the a value, which is the measure of redness or greenness; and the b value, which is the measure of yellowness or blueness. When applying these values to color measurements in citrus fruits, the increase in redness in the juice or concentrate is indicated by an increase in the a values, while the amounts of yellow in the samples are indicated by changes in the b values. By the use of these values, flesh color in grapefruit can be expressed as the ratio of a to b.

Ting and Deszyck (1958) showed graphically (figure 59) that the correlation between the Hunter a/b ratio and lycopene was highly significant ($r = 0.972$). The correlation of the Hunter a value with the lycopene content was also highly significant ($r = 0.938$). In a later paper, Oberbacher *et al.* (1960) reported that, in the Burgundy grapefruit, the correlation coefficient between lycopene content and color expressed as a/b was 0.9924, with $N = 36$.

Ting and Deszyck (1958) also noted that β-carotene was correlated only with the Hunter b value, with a coefficient of $r = 0.357$, which was significant at the 1 per cent level. The correlation of lycopene with β-carotene showed a negative relationship, with a value of $r = -0.227$, with significance at the 5 per cent level. When the ratio of lycopene to β-carotene was correlated with the Hunter ratio a/b, the degree of association proved to be highly significant with $r = 0.770$.

According to the results of Ting and Deszyck, the carotenoid pigment content of grapefruit was definitely related to variety and rootstock. The Ruby Red variety contained a higher concentration of lycopene and β-carotene and consequently a higher internal color than the Thompson pink variety. The Hunter a/b ratio, an index of flesh color, was significantly higher in the Ruby Red variety. By the end of the sampling period (March), the lycopene content of the fruits of both varieties had decreased, but the content of the red variety was considerably more than the amount reported for fruits of the Thompson (figures 60 and 61). With respect to the importance of the 2 rootstocks used in these experiments (rough lemon and sour orange), the fruit of the Ruby Red variety obtained from both stocks had a higher concentration of lycopene

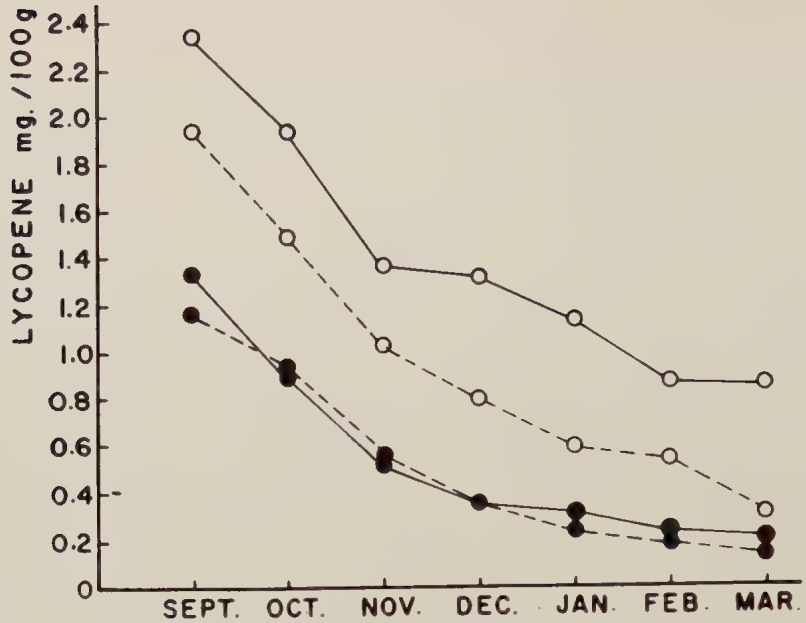


Fig. 60. Seasonal changes of lycopene content of red and pink grapefruit on 2 different rootstocks (half-fruit samples). *Two top curves:* Solid line indicates red on rough lemon; broken line, red on sour orange. *Two lower curves:* Solid line indicates pink on rough lemon; broken line, pink on sour orange. (From Ting and Deszyck, 1958.)

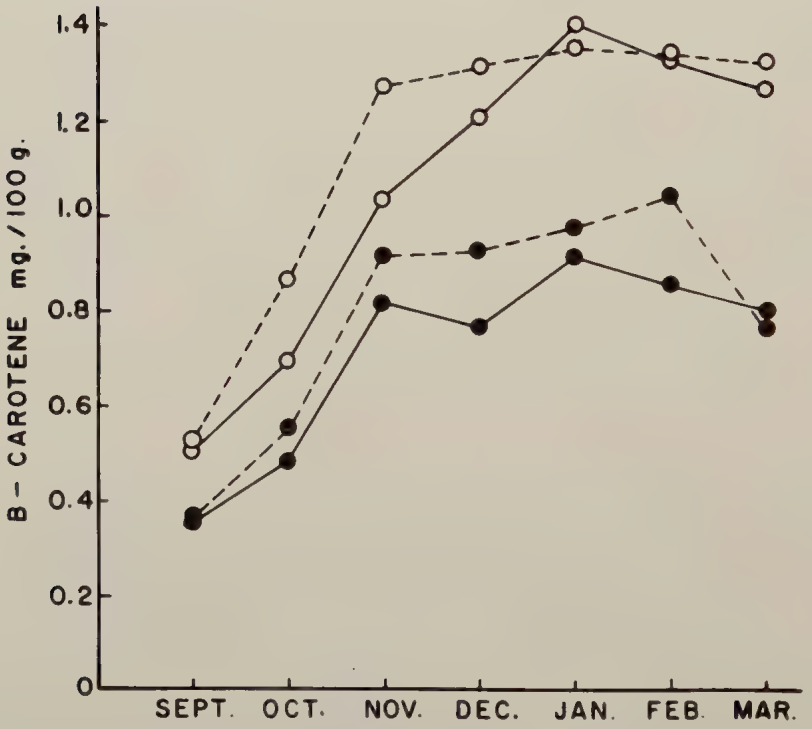


Fig. 61. Seasonal changes of *beta*-carotene content of red and pink grapefruit on 2 different rootstocks (half-fruit samples). *Two top curves:* Solid line indicates red on rough lemon; broken line, red on sour orange. *Two lower curves:* Solid line indicates pink on rough lemon; broken line, pink on sour orange. (From Ting and Deszyck, 1958.)

than fruit of the pink variety harvested from similar rootstocks. Ruby Red grown on rough lemon contained a higher concentration of lycopene than when it was grown on sour orange. The rootstock apparently produced no significant difference in the lycopene content of the pink variety. Sour orange produced fruits with higher β-carotene contents in both the red and pink varieties (figure 60).

These researchers also noted a marked effect of the age of trees on the internal color of Ruby Red and Thompson pink grapefruit grown on rough-lemon rootstock. The color comparisons were made on fruit samples from trees 4 to 6 years old and from others 10 to 14 years old. Both varieties showed more red color in samples from the younger trees. The fruit from the younger trees was significantly higher in the Hunter a/b ratios than that from the older ones. Significant differences in the color of flesh were noted in fruit of the Ruby Red variety on rough-lemon rootstock when the trees were grown in a number of different locations.

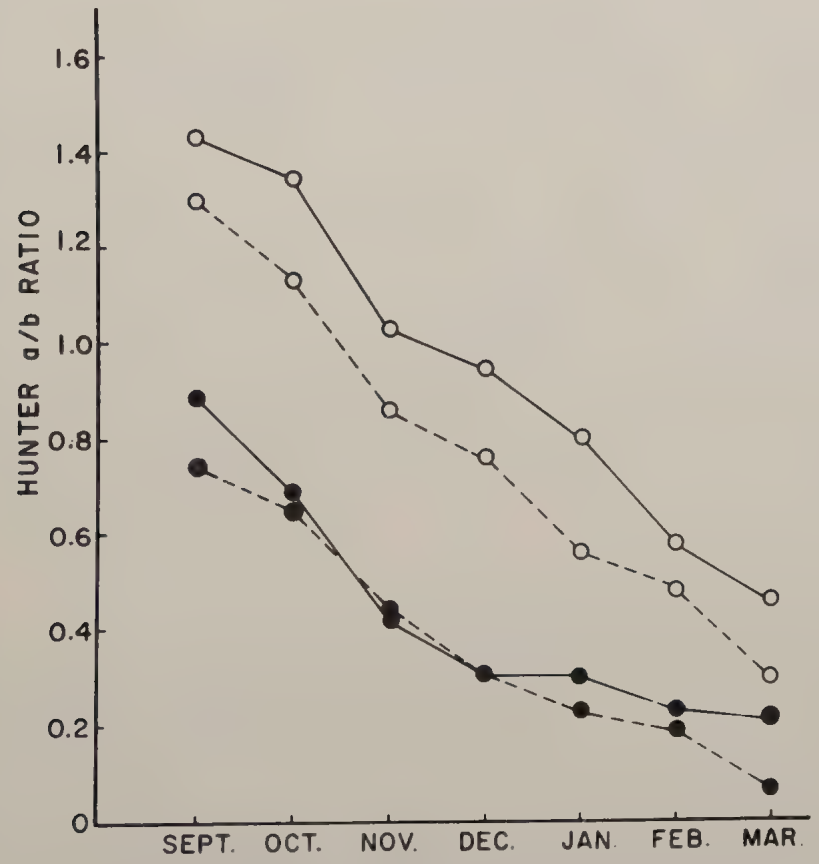


Fig. 62. Seasonal changes of Hunter a/b values of red and pink grapefruit on 2 different rootstocks (purée samples). *Two top curves:* Solid line indicates red on rough lemon; broken line, red on sour orange. *Two lower curves:* Solid line indicates pink on rough lemon; broken line, pink on sour orange. (From Ting and Deszyck, 1958.)

Changes in the color of flesh of red and pink grapefruit were concomitant with the seasonal and compositional changes that occurred during growth and maturation. The Hunter a/b ratios, as indices of internal color, were measured separately for purée samples, and were found to decrease with the advance of the season (figure 62). Although the ratios were higher in the half-fruit samples, the rate of decrease in color was more rapid in the purée samples that were tested.

Fruits of the Ruby Red variety not only had a consistently higher color intensity, but they also showed faster rates of color loss than the Thompson. The fruit produced when either variety was grafted onto sour orange had a tendency to have less internal color. The red color of the fruits, expressed in terms of the Hunter a/b ratios and determined on either half-fruit or purée samples, was highly correlated with the lycopene content.

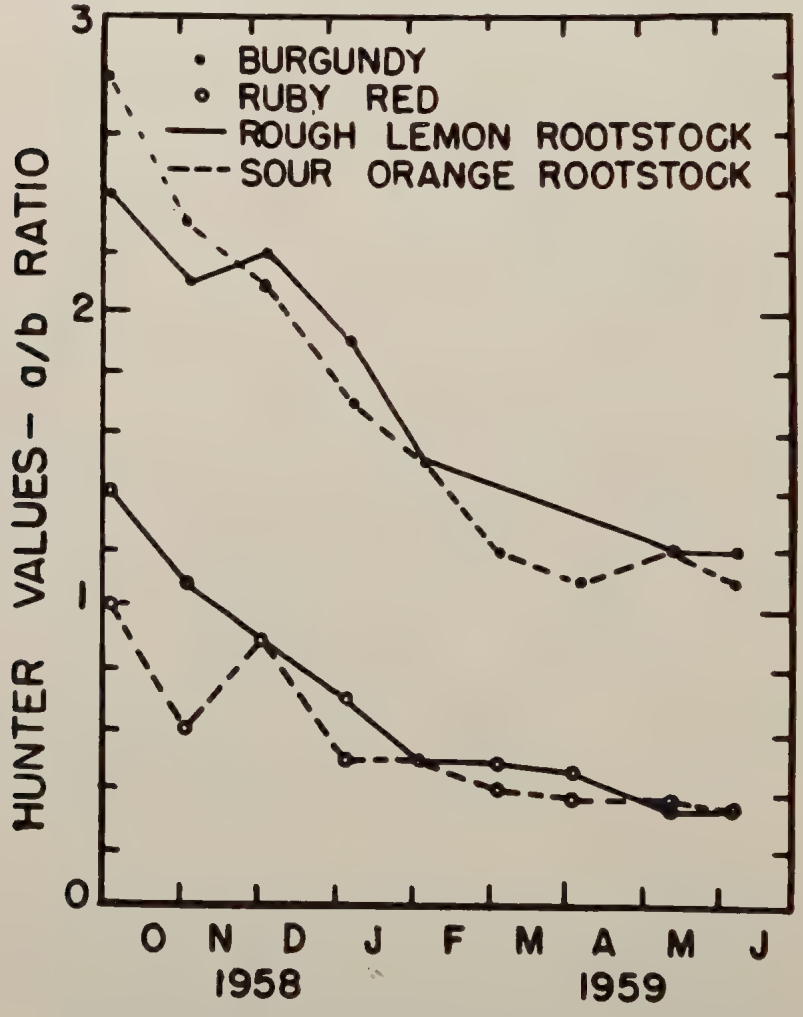


Fig. 63. Monthly changes in the Hunter a/b values of the pulp of Burgundy and Ruby Red grapefruit grown on 2 different rootstocks during the 1958–59 season. (From Oberbacher *et al.*, 1960.)

An analysis of the data in figures 60 and 61 will show the relative changes in lycopene and β-carotene contents of red and pink grapefruit during fruit growth and development. The lycopene contents of these colored varieties decreased with the seasonal advance, while the β-carotene increased to a maximum. During the sampling period, fruits of Ruby Red on rough lemon had a higher concentration of lycopene than those on sour orange. In the Thompson pink variety, however, the 2 rootstocks produced no significant differences in lycopene content. With reference to β-carotene, the situation was quite different: the sour-orange rootstock produced fruits with a higher concentration of this pigment.

Oberbacher *et al.* (1960) found that Burgundy grapefruit, a red-fleshed bud sport of the pink Marsh Seedless variety, was particularly interesting because it had deep-red flesh, and the color persisted in an attractive form throughout the marketing season. They noted that the color was restricted to the flesh, juice vesicles, and segment walls, and that when the variety was propagated from seed, it reverted to the pale type. The color of the Burgundy flesh could be compared with that of the Ruby Red variety by observing the changes in the Hunter a/b ratios of the 2 varieties with the progress of the season (figure 63). At the beginning of the season, the a/b ratio of Burgundy pulp was more than twice as large as that of Ruby Red and, at the end of the season, the a/b ratio of Burgundy was nearly equivalent to the Ruby Red values at the beginning of the season. This relationship also held true for the lycopene content of the pulp, since the color value, expressed as a/b, was highly correlated with the lycopene content. The rates of change of the ratios were about the same for both varieties, as indicated by the average slopes of the curves for the 2 rootstocks. In other words, differences in pulp color and lycopene content of the Burgundy and Ruby Red varieties were maintained during the 9-month sampling period, irrespective of the use of rough-lemon or sour-orange rootstocks.

Other Minor Components

Regarding fiber in edible portions of citrus, Chatfield and McLaughlin (1928) defined it as the loss in weight from incinerating the residue obtained by successive treatments of fat-free dry substances with dilute sulfuric acid and dilute sodium hydroxide. In one of their tables, they quoted other workers as having found an average of 0.3 per cent fiber in the edible portions of several varieties of grapefruit. D. B. Jones (1931) quoted the same figure in his paper on the food values of oranges and grapefruit.

Suberin. The term "suberin," as used here, includes cutin, since by present methods these 2 substances cannot be satisfactorily distinguished. Suberin appears to be more widely distributed than cutin.

Scott and Baker (1947) found that suberin films were not confined to intercellular spaces, but occurred within cells as well. As a result of their studies, they concluded that suberin apparently occurred around and within all living plant cells. Scott (1948) suggested that this finding called for the inclusion of suberin when considering the causes of such processes as transpiration, conduction, abscission, cellulose deposition, and permeability.

As a result of her work on Marsh and Duncan grapefruit, as well as several other kinds of citrus, Gladys King (1947) found deposits on the surfaces of the juice vesicles. Both suberin and one of its principal constituents, phellonic acid, were found in these deposits.

Hydrogen Sulfide. Bartholomew *et al.* (1939) determined small quantities of hydrogen sulfide in the distillates of orange juice.

Nolte *et al.* (1942*b*) reported that orange juice contained a substance that gave a positive test of sulfhydryl. Jansen and Jang (1952) showed that only a small fraction of the sulfhydryl content of fresh citrus juices could be accounted for as hydrogen sulfide.

Kirchner *et al.* (1950), when studying the volatile flavoring constituents of citrus fruits, found small quantities of hydrogen sulfide upon distilling grapefruit and orange juices at about 45° C. (113° F.) and under a vacuum of 40 to 50 millimeters of mercury. On passing nitrogen gas through freshly reamed juices into solutions of lead acetate at room temperature, precipitates of lead sulfide were obtained. These results indicated that free hydrogen sulfide was present in the juices, and that the hydrogen sulfide had not been formed as a result of distillation. Semiquantitative tests yielded 0.45 milligram of hydrogen sulfide per 500 milliliters of Marsh grapefruit juice. The amount of hydrogen sulfide present in citrus juices varied with variety and season. The azide test for mercapto groups was positive on freshly reamed Marsh grapefruit juice and on California Valencia orange juice.

Chapter 3

Composition of Peel, Rag, and Seeds

THE CONCENTRATION of grapefruit growing in relatively few and limited areas and the high percentage of the total production that is now processed create a problem in the disposal of cannery waste, but also provide an opportunity for the recovery of by-products from the waste. In exploring the possibilities for the utilization of by-products, much work has been done on the composition of those parts of the fruit that constitute cannery waste: peel, rag, and seeds. Some components have nutritive values that can be salvaged in dried citrus feed and in molasses. Some are actual or potential ingredients for use in the pharmaceutical and food trades or for industrial purposes. Although several million dollars' worth of products made from citrus wastes are marketed annually, not nearly all the possibilities have been commercially realized.

General Data on Composition

SINCE THE PERCENTAGE of peel varies so widely with variety, climate, and other factors (see chapter 4), and since the number of seeds differs so greatly among varieties, general averages of the amount of waste in grapefruit processing are not too significant. Nevertheless, they may give some idea of the proportions involved. Watt and Merrill (1963) reported 50 per cent (fresh-weight basis) refuse: peel, rag, seeds, membranes around segments, and including handling loss as general averages based on published analyses. Commercial processing methods recover somewhat less of the edible portion of the fruit than that determined by analytical methods, so that the actual percentages of waste in grapefruit canneries will probably always tend to be somewhat higher.

McNary (1947) reported that the peel, rag, and seeds from Florida canneries amounted to about 57 per cent of the over-all weight of the fruit. The data of Shearon and Burdick (1948) on the disposition of materials in juice canning indicate that cannery waste at that time constituted about 61 per cent of the total weight of the grapefruit used, and contained about 69 per cent of the solids, soluble and insoluble. *(See figure 178 of chapter 6, page 509.)*

The proportion of peel in whole, fresh, mature grapefruit was reported as 21 to 26 per cent in Trinidad (Hardy and Rodríguez, 1935); 32 per cent in

TABLE 87

ANALYSIS OF MARSH SEEDLESS GRAPEFRUIT PEEL AND RAG COMBINED (GROUND TOGETHER), AND OF PEEL AND RAG SEPARATELY.*

Constituents	California combined (per cent)	Florida	
		Peel (per cent)	Rag (per cent)
Total soluble solids, in vacuum at 70° C. (158° F.)	22.02	16.71	15.61
Ash	0.70	0.74	0.75
Acids, as citric	.43	.74	.63
Volatile oil (steam-distilled)	.56	.43	....
Ether extract	.23	.28	.16
Crude fiber	2.00	1.71	1.44
Protein (N × 6.25)	1.63	1.13	1.06
Total sugars, as invert	8.68	6.35	6.30
Pentosans	1.31	.83	8.44
Calcium pectate	3.93	3.19	3.56
Naringin	.63	.40	.10

*From Poore (1934). California data are from fresh material; Florida data are from samples that had been sterilized and held in storage.

South Africa (Juritz, 1925); 35 to 39 per cent in Arizona (Hilgeman, 1941); 43 to 47 per cent in California; and 23 to 25 per cent in Florida from "Florida common" grapefruit (Hawkins and Magness, 1920). The last-named authors reported that peel thickness ranged from 4.72 to 6.30 millimeters.

The differences among varieties in the number and weight of seeds have been found to be still greater proportionately. Traub *et al.* (1929) reported the average numbers of seeds per grapefruit in 4 varieties as 3.9 for Marsh, 6.5 for Thompson, 54.7 for Foster, and 63 for Duncan. While, as these figures indicate, there are usually a few seeds in the "seedless" varieties, their percentage of the total weight is negligible. In the seedy varieties, the weight of the seeds as a percentage of the weight of the whole fruit appears to vary in different lots. It was stated to be 2 to 2.5 per cent by Fudge (1938, 1939), and about 4.7 per cent by Nolte and von Loesecke (1940*b*). The latter authors reported that in 1938–39, when the production of seed oil was a new industry, Florida canneries used about 8 million boxes (331,938 tons) of the seedy varieties which, on the basis of 4.7 per cent seeds, they calculated would mean 15,601 tons of seeds.[1] In other grapefruit-growing areas of the United States, seedy varieties comprise a minor percentage of the total production.

Poore (1934) gave general analysis of peel and rag combined from fresh California grapefruit and of peel and rag separately from sterilized and stored Florida grapefruit (table 87).

[1] One ton (2,000 pounds) equals 908 kilograms. One pound equals 0.454 kilogram.

The moisture content of grapefruit peel at maturity is around 80 to 82 per cent, and that of whole, fresh seeds is 55 to 60 per cent. The hulls constitute about 23 per cent, by weight, of the air-dried seeds.

Nolte and von Loesecke (1940*b*) gave the composition of whole, air-dried seeds and seed hulls (table 88).

It may be of interest to compare some of the values given for peel, rag, and seeds in this chapter with the analyses reported for citrus molasses and dried pulp feed in chapter 6.

Sugars in the Peel

CITRUS PEEL contains a large percentage of sugars, especially as the fruit nears maturity. The amounts of soluble carbohydrates and total soluble solids of the peel and pulp of citrus fruits are definitely correlated with the stage of fruit maturity. Climatic differences can produce significant changes in the length of the fruit-crop season, and are therefore highly correlated with the rates of fruit maturity. Grapefruit is outstanding in the differences in time required for fruit to mature under different climatic conditions. Buds on the trees come into full bloom and set fruit at approximately the same time at Riverside and Brawley, California, but about 13 months are required to mature the fruit at Riverside, and only 7 to 8 months at Brawley. The shortening of the time required at Brawley is chiefly owing to the excessive heat units, which accelerate the reactions involved in growth and in the elaboration of food materials.

TABLE 88

ANALYSIS OF WHOLE, AIR-DRIED GRAPEFRUIT SEEDS AND AIR-DRIED HULLS.*

Constituents	In seeds (per cent)	In hulls (per cent)
Moisture	11.86	10.19
Crude fat (ether extract)	30.30	1.17
Protein (N × 6.25)	15.94	4.00
Crude fiber	9.14	39.75
Ash	2.48	2.97
Nitrogen-free extract (by difference)	30.28	....
Silica	0.28	....
Iron and aluminum oxides	.50	....
Calcium	.36	....
Phosphates (P_2O_5)	.56	....
Sodium	.052	....
Potassium	.54	....
Cellulose	.....	32.50
Pentosans	....	16.34

*Adapted from Nolte and von Loesecke (1940*b*).

The mean dry matter and total ash were found to be significantly higher in the peel and pulp of grapefruit samples from Brawley than in those from Riverside. The mean dry-matter content of the juice was about the same in samples from both areas, but the total ash was much higher in the juice samples from Brawley.

Poore (1934) found 8.68 per cent total invert sugars (wet-weight basis) in fresh California grapefruit whole peel and rag that had been ground together. He analyzed Florida peel and rag separately, and found 6.35 per cent total invert sugar in the peel and 6.30 per cent in the rag (table 87). The California and Florida values are not comparable because the samples from Florida had been sterilized and kept in storage before being analyzed.

Haas and Klotz (1935) found that the dry matter of grapefruit peel contained 28.77 per cent total sugars in the stem half and 27.89 per cent in the stylar half.

Harvey and Rygg (1936*c*) determined the sugar content of the whole peel of Marsh grapefruit grown in 2 widely separated areas in southern California—Corona-Fontana and Oasis. The determinations were made in the 1933–34 season. The results are shown in table 89, in which the sugar values are based on the dry weight of the peel. These workers also tested for differences between halves of the Marsh peel in the same areas. Their average results for sugars are shown in table 90. They stated that the differences between the 2 halves were surprisingly small when compared with those for Washington Navel and Valencia orange peels. The average differences for the totals of all solids (not shown in the table) were 3.4 per cent in the Corona-Fontana area and only 1.3 per cent in the Oasis area. The corresponding differences for the Washington Navels and Valencias in the Corona-Fontana area were 13 and 11 per cent, respectively.

Sinclair and Crandall (1949*a*) determined the carbohydrates of Marsh grapefruit peel (table 91). They made 5 determinations each on 2 lots of fruit, one that had been fumigated and the other oil-sprayed. The peel from 50 to 100 grapefruit was used in each of the 10 tests. The mean amounts of reducing sugar and sucrose, based on the dry weight of the peel, were 24.35 and 15.94 per cent for the fumigated fruits and 25.12 and 16.16 per cent for the oil-sprayed ones. Thus, the total soluble sugars in the peel were 40.29 and 41.28 per cent, respectively. They also reported that the alcohol-soluble solids of the peel amounted to 66.31 and 67.35 per cent of the dry matter for fumigated and oil-sprayed fruit, respectively. Similar data for the alcohol-insoluble solids amounted to 33.69 and 32.65 per cent.

Ting and Deszyck (1961), applying the procedures of Sinclair and Crandall (1949*a*) and of Sinclair and Jolliffe (1960), extracted Marsh grapefruit peel with 80 per cent ethyl alcohol to separate the peel constituents into their alcohol-soluble and alcohol-insoluble fractions. They made extended analy-

TABLE 89

REDUCING AND SUCROSE SUGARS NOTED IN DIFFERENT MONTHS IN THE PEEL OF MARSH GRAPEFRUIT GROWN IN 2 AREAS OF CALIFORNIA.*

(Dry-weight basis.)

Dates	Reducing sugar (per cent)	Sucrose (per cent)	Total sugar (per cent)
CORONA-FONTANA: AVERAGES OF 16 DETERMINATIONS			
1933:			
March 19	26.55	8.10	34.65
April 30	26.09	9.53	35.62
June 11	25.12	11.67	36.79
July 23	23.06	16.30	39.36
September 6	23.81	13.63	37.44
OASIS: AVERAGES OF 8 DETERMINATIONS			
1933:			
November 6	14.23	7.56	21.79
December 11	18.55	9.24	27.79
1934:			
January 15	27.04	7.69	34.73
March 1	22.69	9.95	32.64
April 9	20.38	14.00	34.38

*Adapted from Harvey and Rygg (1936c).

TABLE 90

REDUCING AND SUCROSE SUGARS IN THE PEEL FROM 2 HALVES OF MARSH GRAPEFRUIT GROWN IN THE CORONA-FONTANA AND OASIS AREAS OF CALIFORNIA.*

(Dry-weight basis.)

Fruit parts	Reducing sugar (per cent)	Sucrose (per cent)	Total sugar (per cent)
CORONA-FONTANA (1933)			
Stem end	25.23	12.18	37.41
Stylar end	24.62	11.50	36.12
OASIS (1933–34)			
Stem end	20.73	9.04	29.77
Stylar end	20.42	10.33	30.75

*Adapted from Harvey and Rygg (1936c).

TABLE 91

Soluble Carbohydrates of Mature Marsh Grapefruit Peel.*

Sample numbers	Moisture (per cent of fresh weight)	Alcohol-insoluble solids (per cent of dry weight)	Alcohol-soluble solids (per cent of dry weight)	Water-soluble sugars (as glucose) in alcoholic extract (per cent of dry weight)			Total sugars (per cent of alcohol-soluble solids)
				Reducing	Sucrose	Totals	
Fumigated Fruit							
1	81.46	33.47	66.53	24.12	15.89	40.01	60.14
2	81.33	33.42	66.58	24.08	15.25	39.33	59.07
3	81.52	33.69	66.31	23.97	16.68	40.65	61.30
4	81.40	33.84	66.16	24.19	16.04	40.23	60.81
5	81.46	34.04	65.96	25.38	15.84	41.22	62.49
Means	81.43	33.69	66.31	24.35	15.94	40.29	60.76
Oil-sprayed Fruit							
1	82.23	32.95	67.05	24.87	17.46	42.33	63.13
2	82.01	32.65	67.35	24.53	15.57	40.10	59.55
3	82.13	32.73	67.27	25.98	16.34	42.32	62.90
4	82.11	32.26	67.74	25.31	16.24	41.55	61.34
5	82.23	32.65	67.35	24.91	15.17	40.08	59.50
Means	82.14	32.65	67.35	25.12	16.16	41.28	61.28

*From Sinclair and Crandall (1949*a*).

ses of the carbohydrates of these 2 fractions, using the newer methods of paper chromatography. They noted that the seasonal changes of alcohol-insoluble solids decreased from 68.6 per cent to 49.8 per cent of the dry weight during a sampling period from August through January (table 92). (A similar decrease had been noted for Valencia oranges by Sinclair and Jolliffe (1958) for both the alcohol-insoluble and acetone-insoluble fractions.) In sampling Valencia oranges from October to the following September, the insoluble solids for both the 80 per cent alcohol and the acetone extractants decreased from 70 to about 50 per cent of the dry weights. In their final sample of the season, Ting and Deszyck reported that the alcohol-insoluble solids fraction contained 52.4 per cent of its weight as pectic substances, 11.7 per cent as hemicellulose, 30.8 per cent as cellulose, and 5.1 per cent was undetermined (table 92). Their data showed that a seasonal increase in the alcohol-soluble solids fraction occurred, and consequently the total soluble carbohydrates of the peel increased, during the sampling period, from 26.2 to 39 per cent of the dry weight (table 93). This was what should have been expected, owing to the fact that as the soluble carbohydrates increased in the fruit during growth and maturation, they logically should have appeared in the alcohol-soluble fraction. In the January sample, the alcohol-soluble fraction contained 13.5 per cent glucose, 13.5 per cent fructose, and 11.9 per cent sucrose, all calculated on the percentage of dry weight of the peel.

The data in table 94 show the relative amounts of polysaccharides in the alcohol-insoluble fractions of various citrus varieties, including Marsh grapefruit. Although Ting and Deszyck (1961) did not fractionate the alcohol-insoluble solids, they did hydrolyze each fraction, and determined the characteristic monoses in each hydrolysate. Polygalacturonic acids and cellulose glucosan were the most abundant polysaccharides of the peel. The glucosan fraction was greater in Marsh grapefruit than in the other citrus varieties. The major sources of the araban and galactan may have been the polygalacturonic acids. Since the individual polysaccharides were not quantitatively estimated, each sugar that was determined was assumed to be derived from a single polysaccharide. Thus, arabinose was assumed to be derived from araban which, when derived from the alcohol-insoluble solids complex, was a fairly valid assumption.

Acids and pH of the Peel

MARSH GRAPEFRUIT PEEL grown in the Corona-Fontana area of California was found by Harvey and Rygg (1936*c*) to have, on March 19, 1933, a pH of 6.31 and, on September 6, a pH of 6.24. During this period, the pH in the peel changed only slightly, but at the third sampling on June 11, 1934, an increase to 6.53 occurred. Similar determinations made on grapefruit peel from Oasis, a California area located farther inland, showed that the first sample, taken

TABLE 92

PERCENTAGE DISTRIBUTIONS OF VARIOUS FRACTIONS IN THE ALCOHOL-INSOLUBLE SOLIDS OF MARSH GRAPEFRUIT PEEL.*

Times of sampling	Alcohol-insoluble solids (per cent of dry weight)	Fractions (per cent of alcohol-insoluble solids)			Not accounted for
		Pectic substances	Hemicellulose	Cellulose	
August	68.6	44.0	9.4	39.5	7.1
September	66.6	42.5	12.1	40.4	5.2
October	56.9	42.1	12.4	40.3	5.2
November	54.9	48.3	11.4	35.2	5.1
December	51.1	46.1	10.2	37.7	6.0
January	49.8	52.4	11.7	30.8	5.1

*From Ting and Deszyck (1961).

TABLE 93

SEASONAL DIFFERENCES IN THE SUGAR COMPOSITION OF MARSH GRAPEFRUIT PEEL (1957–58 SEASON).*

Times of sampling	Dry weight (per cent)	Alcohol-soluble solids (per cent)	Percentages (dry weight)			Total sugars (per cent)
			Glucose	Fructose	Sucrose	
August	17.30	31.4	11.7	10.4	4.0	26.2
September	16.74	33.4	8.2	8.3	9.0	25.5
October	18.36	43.1	7.2	6.8	16.2	30.3
November	19.31	45.1	8.5	7.2	20.9	36.7
December	19.71	48.9	11.6	12.8	14.3	38.7
January	17.91	50.2	13.5	13.5	11.9	39.0

*From Ting and Deszyck (1961).

TABLE 94

POLYSACCHARIDES AS PERCENTAGES OF THE ALCOHOL-INSOLUBLE SOLIDS OF CITRUS PEEL.*

(Mean values for the entire season.)

Fruit varieties	Araban	Galactan	Xylan	Glucosan	Polygalact-uronic acids	Other uronic acids	Totals†
Hamlin orange	9.3	6.1	2.6	20.8	23.2	0.7	62.7
Pineapple orange	7.0	4.8	2.2	15.5	23.5	.4	53.4
Valencia orange	9.7	5.3	2.1	14.8	23.6	.5	56.0
Marsh grapefruit	9.9	4.9	2.7	27.9	23.4	1.2	70.0

*From Ting and Deszyck (1961).

†Factors used to convert monosaccharides to polysaccharides were respectively 0.88, 0.90, and 0.92 for pentoses, hexoses, and hexuronic acids.

November 6, 1933, had a pH of 5.74, and the last one, taken April 9, 1934, had a pH of 6.46. These differences were much larger than those recorded for the Corona-Fontana region.

The same workers also reported the pH determinations for different portions of the Marsh peel. As may be seen in table 95, the stem and stylar ends of the peel samples had approximately the same pH values. With respect to the flavedo and albedo portions, the flavedo possessed the higher pH values.

The relationship of pH to the acid constituents of the peel sap can be elucidated by following the resistance to changes in pH upon the addition of increments of standard acid or alkali to a known amount of sap. The sap, in common with all plant extracts, is to some degree capable of resisting changes in pH upon the addition of strong acids or bases.

Sinclair and Eny (1947) determined the titration curve of the peel sap of grapefruit. The sap was prepared by cutting the fresh whole peel into small pieces, placing them in covered glass jars, and freezing for 48 to 60 hours at –25° C. (–13° F.). After thawing, the peel was quickly ground through a galvanized meat chopper. The juice was extracted from this ground mass by means of a hydraulic press at a pressure of 20,000 pounds per square inch (9,080 kilograms; 6.45 cm^2). This liquid was used for determining the titration curve.

It was important to determine the effect of the soluble pectic substances in the centrifuged sap on the shape of the buffer curve. The titration curves of normal sap (curve *A*) and of sap with pectin removed (curve *B*) are shown for grapefruit peel in figure 64. Although the 2 curves are nearly parallel, they are distinctly different. The alcohol used for precipitating the pectic

TABLE 95

THE pH OF DIFFERENT PORTIONS OF THE PEEL OF MATURE MARSH GRAPEFRUIT GROWN IN 2 AREAS OF CALIFORNIA.*

Localities and years	Portions of peel	pH (mean values)	Ranges in pH
Corona-Fontana (1933)	Stem end	6.45	6.25–6.66
	Stylar end	6.40	6.29–6.59
Oasis (1933–34)	Stem end	6.24	5.78–6.79
	Stylar end	6.22	5.70–6.61
Corona-Fontana (1933)	Flavedo	6.72	6.52–6.94
	Albedo	6.14	5.96–6.30
Oasis (1933–34)	Flavedo	6.54	5.88–7.07
	Albedo	5.93	5.60–6.34

*Adapted from Harvey and Rygg (1936c).

materials probably precipitated some of the soluble buffer salts also, and thus caused an increase in acidity and lowered the position of curve *B* on the graph. The amount of free acid in this sap is represented by the portion of the curve between zero and the milliequivalents of sodium hydroxide required to bring the system to a pH of 8.2. It can be seen that the concentration of free acid, organic and inorganic, was relatively small in this experiment.

The positions of the curves on the graph with respect to buffer properties were influenced by the concentration of free acid in the sap. In other words, the buffer capacity of a solution is governed by the concentration of the components. As the sap of grapefruit peel is not highly buffered, its titration curves at different dilutions will have different slopes.

The titration curves of grapefruit peel saps are vastly different from those of the juice of the pulp. The peel of grapefruit contains a comparatively small amount of organic acids and their salts; consequently, the pH values of the peel are high, and these systems have only a slight buffer capacity. The pulp juice of citrus fruits, on the other hand, contains larger amounts of organic acids and their salts, and has lower pH values and higher buffer capacities.

Organic Acids Present. Sinclair and Eny (1947) determined the ether-soluble organic acids of dried grapefruit peel (table 96). Although the data are reported on a fresh-weight basis, actual determinations were made on the ether extract of dried peel. The distinctive features of the table are the comparatively low concentrations of citric acid and the relatively high concentrations of malic and oxalic acids in the samples (compare with acids in

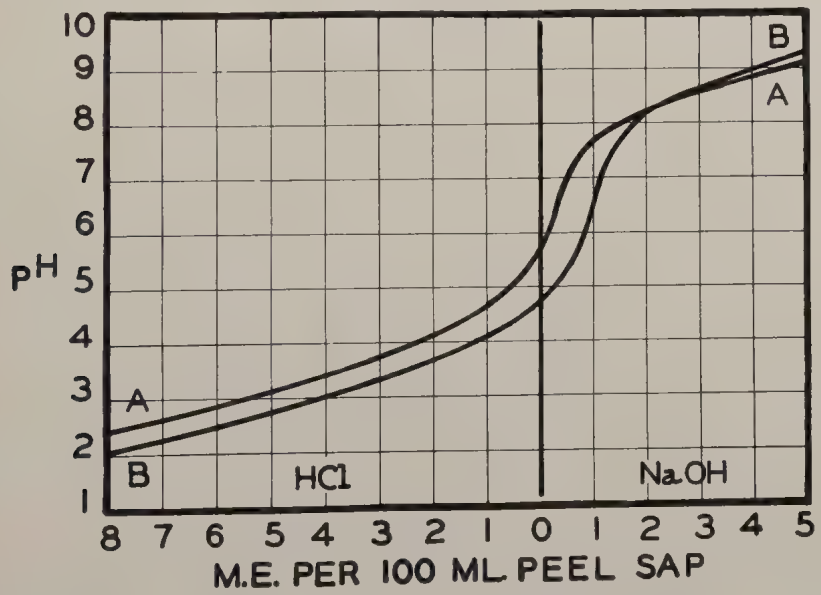


Fig. 64. Titration curves of grapefruit-peel sap. *A*, normal; *B*, pectin removed. (From Sinclair and Eny, 1947.)

TABLE 96

Ether-soluble Organic Acids of Grapefruit Peel.*

Sample numbers	Moisture (per cent)	pH of sap	Ether-soluble organic acids									
			Total acids, as citric (pH 7.8–2.6)		Citric acid		Malic acid		Oxalic acid		Total citric, malic, and oxalic acids	
			mg/gm†	meq/gm‡	mg/gm	meq/gm	mg/gm	meq/gm	mg/gm	meq/gm	mg/gm	meq/gm
1	80.18	....	2.88	0.045	0.32	0.005	1.27	0.019	0.86	0.019	2.75	0.043
2	80.56	....	2.75	.043	.32	.005	1.47	.022	.68	.015	2.69	.042
3	82.39	5.70	2.37	.037	.38	.006	1.34	.020	.50	.011	2.37	.037
4	81.52	5.65	2.88	.045	.38	.006	1.68	.025	.59	.013	2.82	.044

*Adapted from Sinclair and Eny (1947).
†Milligrams of acid (as citric) per gram of fresh peel.
‡Milliequivalents per gram of fresh peel.

grapefruit juice). By extracting dried peel with ether at pH 1, the insoluble oxalates were made soluble and were readily removed from the tissue by the solvent. An abundance of calcium in the peel indicates that any oxalic acid occurring in the peel will be immediately converted to the insoluble calcium salt; but that is true only if an excess of calcium is present in the peel over that required to form calcium pectate. The fact that only a trace of oxalic acid appeared in the peel sap was evidence that sufficient calcium was present to convert the oxalic acid into insoluble calcium oxalate. The high calcium content of the peel (0.64 per cent, dry-weight basis) in comparison with that of the pulp and juice (0.26 and 0.07 per cent, respectively, dry-weight basis) was certainly highly correlated with the amount of insoluble oxalate. In fact, calcium and magnesium were the only inorganic cations that existed in the peel in concentrations sufficiently high to form crystalline insoluble salts with the organic acids.

Ting and Deszyck (1959) reported that quinic acid was the chief organic acid in the peel of citrus fruits, and that it had an equivalent weight approximately 3 times that of malic or citric acids. The concentration of quinic acid present in mature Marsh grapefruit amounted to 233 milligrams per 100 grams of fresh peel, and 87 milligrams per 100 grams of fresh tissue. The values reported for Marsh grapefruit peel were significantly higher than similar values reported for other varieties of citrus fruits (Pineapple orange, Dancy tangerine, Correggio lemon, and Persian lime).

Proteins and Amino Acids

GRAPEFRUIT PEEL AND RAG were ground together by Poore (1934) in California. He found that the mixture contained 1.63 per cent protein. He also ground sterilized and stored Florida grapefruit peel and rag separately, and analyzed them for protein. The peel contained 1.13 per cent and the rag, 1.06 per cent. (See table 87 of this chapter.)

Sinclair and Crandall[2] determined the proportion of total nitrogen of grapefruit peel that occurred in the alcohol-insoluble and alcohol-soluble fractions. The data in table 97 show that an average of 48.62 per cent of the total nitrogen was soluble in 80 per cent alcohol. The amount of nitrogen that remained in the alcohol-insoluble fraction was attributed to the protein content of the whole peel. The alcohol-soluble nitrogen came from the amide, basic, and nitrate nitrogen and from the α-amino acids and peptides. The nitrate nitrogen amounted to only 0.08 per cent of the dry weight of the whole peel.

Townsley *et al.* (1953) determined the total nitrogen extractable with 70 to 80 per cent alcohol from various tissues of mature grapefruit and other citrus types (see table 63 of chapter 2). The flavedo and vesicle tissues of

[2] W. B. Sinclair and P. R. Crandall. Unpublished data.

grapefruit contained 15.87 and 15.16 milligrams of nitrogen per 100 milliliters of extract, respectively, and the albedo contained 7.66 milligrams of nitrogen on the same basis. The seeds contained the greatest concentration of alcohol-soluble nitrogen (22.62 milligrams per 100 milliliters of extract) reported for grapefruit.

Townsley and associates, using oranges but not grapefruit, also isolated a protein from the peel that contained 4.35 per cent total nitrogen, indicating its contamination with 65 to 70 per cent nonprotein material. In purifying the peel protein, the filtrate that remained after the protein was precipitated at pH 4.7 and filtered was bright yellow. The color intensity apparently was not affected by repeated precipitation, and was not removable by washing the protein with water. The peel protein was associated with a substance which may or may not have been a chemical combined with the protein, and which was quite insoluble in the range of pH 4.7. Tests indicated that this impurity was carbohydrate in nature.

Sinclair *et al.* (1935), in their investigations of orange-pulp proteins, suggested that a similar carbohydrate material was pectin. The precipitation of pectin with the protein may have been responsible for the occlusion of other impurities, such as those responsible for the color and also for the fluorescence found in the barium hydroxide hydrolysis.

TABLE 97

NITROGEN IN WHOLE PEEL AND IN ALCOHOL-INSOLUBLE SOLIDS OF GRAPEFRUIT PEEL.*

Material used	Nitrogen content (per cent)				
	Alcohol-insoluble solids basis	Dry-weight basis	Amount soluble in 80 per cent alcohol		Nitrate nitrogen (dry-weight basis)
			Dry-weight basis	Proportion of the total N	
Sample 1:					
Whole peel	....	0.91	....	....	0.08
Alcohol-insoluble solids (a)	1.28	.46	0.45	49.45	....
(b)	1.30	.47	.44	48.35	....
(c)	1.30	.47	.44	48.35	....
Sample 2:					
Whole peel	....	0.90	....	....	0.08
Alcohol-insoluble solids (a)	1.33	.47	0.43	47.78	....
(b)	1.30	.46	.44	48.89	....
(c)	1.28	.46	.44	48.89	....

*W. B. Sinclair and P. R. Crandall (unpublished).

Cameron *et al.* (1936) determined the seasonal changes in the nitrogen content of California oranges, grapefruit, and Eureka lemons. The rate of increase in total nitrogen was higher for grapefruit than for the other citrus types studied. They noted that the total nitrogen content per fruit was significantly correlated with the increase in dry weight during growth and maturation. The nitrogen in different component parts of both Valencia and navel oranges increased during fruit growth. In the juice of navel oranges, it increased at a faster rate than in either the rind or the pulp. (The nitrogen content of the fruit will continue to increase if it is allowed to remain on the tree long after peak maturity.)

Appleman and Richards (1939), in determining the distribution of total nitrogen in various tissues of California Valencia oranges, reported that the rind and juice contained 44.8 and 41.1 per cent, respectively, of the total nitrogen in the fruit, while the rag had the remaining 14.1 per cent. With the continued seasonal increase in nitrogen, a corresponding increase in the syntheses of amino acids and proteins also should have continued to occur in the fruit as long as it was attached to the tree.

Goren and Monselise (1964) recorded the results of further studies on the seasonal changes in total and protein nitrogen of citrus fruits, working with the Shamouti orange. Although their data were obtained from orange fruits, they would doubtless be highly applicable to seasonal changes of nitrogen in grapefruit. The time-course curves for changes in total and protein nitrogen show that when the values were expressed as percentages of dry weight and as milligrams per fruit, the slopes and directions of the curves were entirely different (figure 65). On the first sample, the young fruit had 2.1 per cent total nitrogen and 1.4 per cent protein nitrogen on a dry-weight basis. To express these values on an individual fruit basis, the total and protein nitrogen amounted to 3 and 2 milligrams per fruit, respectively. Both the total and protein nitrogen decreased during growth and development of the fruit as percentages of dry weight. In the last fruit sample (January), the total and protein nitrogen had decreased to 1.2 and 0.8 per cent of their respective dry weights.

It should be pointed out that the dry weight or dry matter per fruit increases with fruit growth.

In agreement with the results of Cameron *et al.* (1936), Goren and Monselise further found that the amount of nitrogen per fruit increased with fruit growth and development as long as the fruit remained attached to the tree.

At the end of the experiment, the fruit contained (juice excluded) 300 milligrams of total and 175 milligrams of protein nitrogen, a relationship which showed that 58 per cent of the total nitrogen was derived from protein material.

Seeds

THE SEED COUNT of citrus fruits increases irregularly but persistently with fruit size—a peculiarity shared with many other multiseeded fruits. Cameron *et al.* (1960) reported a significant correlation between fruit size and seed number in Valencia oranges, but this association appears to have been limited chiefly to the smaller fruit-size classes. In the larger classes, the seed count not only leveled off, but decreased. The larger fruits which contained relatively few seeds may have originated under atypical conditions which permitted them to develop into larger sizes even though they contained low seed counts. Cameron and his co-workers obtained part of their data from Valencia oranges taken from trees that had been exposed to open pollination from other varieties, but also recorded additional data from trees exposed only to Valencia pollen, which resulted in fruit containing a positive increase in seed number with increase in size. They drew attention to the fact that in Valencia and related fruits, seed number is related to fruit size, but that the Valencia is parthenocarpic to some degree; consequently, the presence and number of seeds constitute only a portion of many factors that may relate to fruit development.

Krezdorn (1967) reported that increased seediness resulted in increased fruit size for many kinds of citrus, including Marsh grapefruit (table 98). (This relationship, while not shown in the table, also held for the Duncan variety.) The sizes were classed as small, medium, and large. The "Page" is

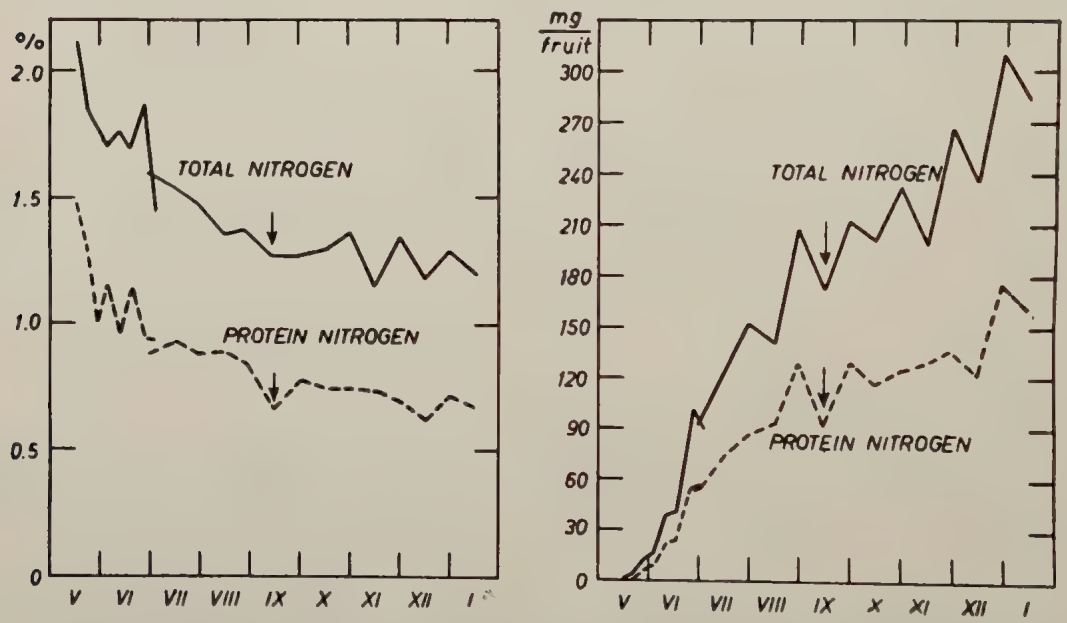


Fig. 65. Seasonal changes in total and protein nitrogen of developing Shamouti oranges, expressed as percentages of dry weight and milligrams per fruit. An arrow on a curve indicates the month after which juice weight was not included in the curve. (From Goren and Monselise, 1964.)

a variety that produces undesirably small fruit, especially in arid climates, and its seedy fruit is larger than that of the seedless and few-seeded variants. Since it is highly parthenocarpic, and thus may produce many small, seedless fruits, cross-pollination tends to increase the over-all sizes of fruit produced by a tree, but probably not enough to overcome the size problem. The regression coefficient of fruit diameter on seed number was highly significant ($r = 0.7706$).

Oils. The seed oils of citrus fruits are composed of true fats, which are usually defined as the glycerol esters of the higher fatty acids. When seedy varieties of grapefruit are processed, seed oils contribute to the nutritive values of feeds made from the waste. Florida canneries have also found it profitable to extract the oil from the seeds and market it separately. The more detailed studies of seed oils have been made with the latter purpose.

TABLE 98

MEAN NUMBER OF SEEDS PER FRUIT IN EACH OF 3 SIZE CLASSES OF SEVERAL TYPES OF CITRUS.*

Varieties	Experiment numbers	Fruit size classes†		
		Small	Medium	Large
Hamlin orange	1	1.2	2.3	3.9
	2	1.3	2.1	3.2
	3	1.5	2.6	3.0
Parson Brown orange	1	5.2	7.5	10.0
	2	6.9	8.7	11.2
Pineapple orange	1	7.8	9.4	12.2
	2	9.4	13.0	15.5
	3	10.1	13.0	15.4
Valencia orange	1	2.1	3.2	5.0
	2	2.6	4.0	6.4
	3	2.3	3.7	5.9
Marsh grapefruit	1	2.4	6.2	6.2
	2	2.1	4.5	6.4
Dancy tangerine	1	8.9	11.6	15.5
	2	10.9	12.4	18.0
Robinson tangerine-tangelo	1	3.4	8.9	16.2
"Page" (see text)	1	0.6	9.2	15.7

*From Krezdorn (1967).
†Each entry is the mean or average number of seeds per fruit in the given size class.

Jamieson *et al.* (1930) found that grapefruit seeds contained about 30 per cent oil on a dry-weight basis. They found the percentage composition of this semidrying oil to be: 20.5 olein, 50.1 linolein, 20.1 palmitin, 7.6 stearin, 0.1 lignocerin, and 0.7 unsaponifiable acids.

Nolte and von Loesecke (1940*b*) reported 28 to 35 per cent oil in air-dried seeds, shown earlier in table 88. Table 99 records the components and values they found for press cake after the oil had been removed. They also gave the various comparative compositions and physical characteristics for unrefined, bleached, and "wintered" grapefruit-seed oil; see table 100. The unrefined oil was reddish brown and had a pleasant, nutlike flavor, but was extremely bitter. The commercial oil was cloudy at 15°, pasty at 4°, buttery at – 4°, and solid at – 10° C. In moderately thick layers, the bleached product was light straw color. "Wintered" oil was refined and bleached oil that had been chilled at 1° C. (34° F.) and put through a small hand press, which operation did not press all the oil from the solidified stearin. This oil was light yellow and bitter, owing to the presence of limonin.

Dunn *et al.* (1948) stated that 25 per cent of the fatty acids of West Indian citrus fruits were saturated, and that grapefruit seeds contained 40 per cent fatty acids of good quality.

TABLE 99

COMPOSITION OF THE PRESS CAKE OBTAINED IN THE PREPARATION OF GRAPEFRUIT-SEED OIL.*

Constituents	Per cent	Constituents	Per cent
Moisture	3.43	SiO_2	0.081
Ash	4.04	S	.088
Nitrogen as NH_3	4.21	Ca	.35
Nitrogen as protein	21.60	Mg (MgO)	.39
Crude fat (ether extract)	13.95	NaCl + KCl	2.48
Crude fiber	26.50	Phosphates (P_2O_5)	.55
		Fe	.0014

*From Nolte and von Loesecke (1940*b*).

TABLE 100

THE COMPARATIVE COMPOSITION AND PHYSICAL CHARACTERISTICS OF 3 GRAPEFRUIT-SEED OILS.*

Oil	Specific gravity 25°/25° C.	Refractive index	Acid value	Saponification value	Iodine number	Unsaponifiable matter (per cent)
Commercial	0.9197	1.4698	0.95	193.0	100.9	0.48
Bleached	.9179	1.4688	.17	197.5	101.7	.38
"Wintered"	.9199	1.4700	.26	192.2	109.2	....

*Adapted from Nolte and von Loesecke (1940*b*).

TABLE 101

PHYSICAL AND CHEMICAL PROPERTIES OF GRAPEFRUIT- AND SHADDOCK-SEED OILS PRODUCED IN FLORIDA.*

Varieties of fruit	Dates processed	Refractive indices n_D^{25}	Iodine absorption numbers	Fatty acids (per cent)				
				Palmitic	Stearic	Oleic	Linoleic	Linolenic
Excelsior	8/29/60	1.4689	98.3	34.6	2.2	22.2	38.0	3.0
Excelsior	10/26/60	1.4689	99.6	34.5	3.6	20.5	36.2	5.2
Excelsior	1/3/61	1.4692	99.7	35.8	3.6	22.8	34.2	3.6
Excelsior	2/10/61	1.4695	101.0	31.1	5.0	22.5	37.2	4.2
Excelsior	3/23/61	1.4695	97.8	34.4	4.8	21.2	34.8	4.8
Duncan	8/29/60	1.4690	96.8	35.6	1.6	20.6	39.2	3.3
Duncan	10/26/60	1.4691	100.0	36.7	3.6	20.0	34.8	4.9
Duncan	1/3/61	1.4692	100.0	35.4	2.7	22.4	36.5	3.0
Duncan	2/10/61	1.4693	100.0	31.1	4.4	20.7	38.7	5.1
Duncan	3/23/61	1.4696	99.5	35.9	3.4	20.6	35.4	4.7
Walters	8/29/60	1.4690	96.0	35.0	2.9	21.1	37.6	3.4
Walters	10/26/60	1.4691	98.1	37.5	3.7	18.6	37.4	2.8
Walters	1/3/61	1.4692	98.9	35.2	3.7	22.5	34.6	4.0
Walters	2/10/61	1.4692	100.0	33.0	3.8	20.8	37.7	4.7
Walters	3/23/61	1.4695	99.6	34.8	3.8	20.6	36.3	4.5
Marsh	10/3/60	1.4691	98.5	34.5	3.6	22.9	35.4	3.6
Marsh	1/3/61	1.4692	98.8	34.7	2.8	22.2	35.4	4.9
Marsh	2/10/61	1.4693	100.0	35.0	3.1	20.0	37.3	4.6
Marsh	3/23/61	1.4696	101.0	36.4	3.1	19.5	36.4	4.6
Shaddock	8/29/60	1.4690	97.5	30.5	3.2	24.7	38.0	3.6
Shaddock	3/23/61	1.4692	99.4	26.0	4.9	27.2	37.0	4.9

*From Hendrickson and Kesterson (1961).

Hendrickson and Kesterson (1961) investigated the physical and chemical properties of grapefruit- and shaddock-seed oils produced in Florida by the use of improved gas chromatographic procedures for the qualitative and quantitative determinations of fatty acids. In the varieties on which they reported, the refractive indices of the oils increased with fruit maturity (table 101). At a given processing date, the refractive indices of the seed oils of these varieties were not significantly different. The iodine absorption number of an oil, as shown in the table, is an indication of the amounts of unsaturated fatty acids it contains in the free and glyceride forms. This property can serve as a basis for their quantitative determination. The iodine number of an oil is also an indication of whether the type of oil is drying, semidrying, or nondrying. The higher the iodine number of an oil, the greater is the degree of its unsaturation and consequently of its drying property. The iodine numbers of the grapefruit- and shaddock-seed oils were found to be remarkably similar. The values were high enough to indicate that the degrees of unsaturation of the fatty acids in these oils might show them to be of therapeutic value when used in diets.

The fatty acid compositions of the grapefruit- and shaddock-seed oils are shown in table 101. Approximately one-third of the fatty acid content was composed of linoleic acid, and a total of about two-thirds, consisting of oleic,

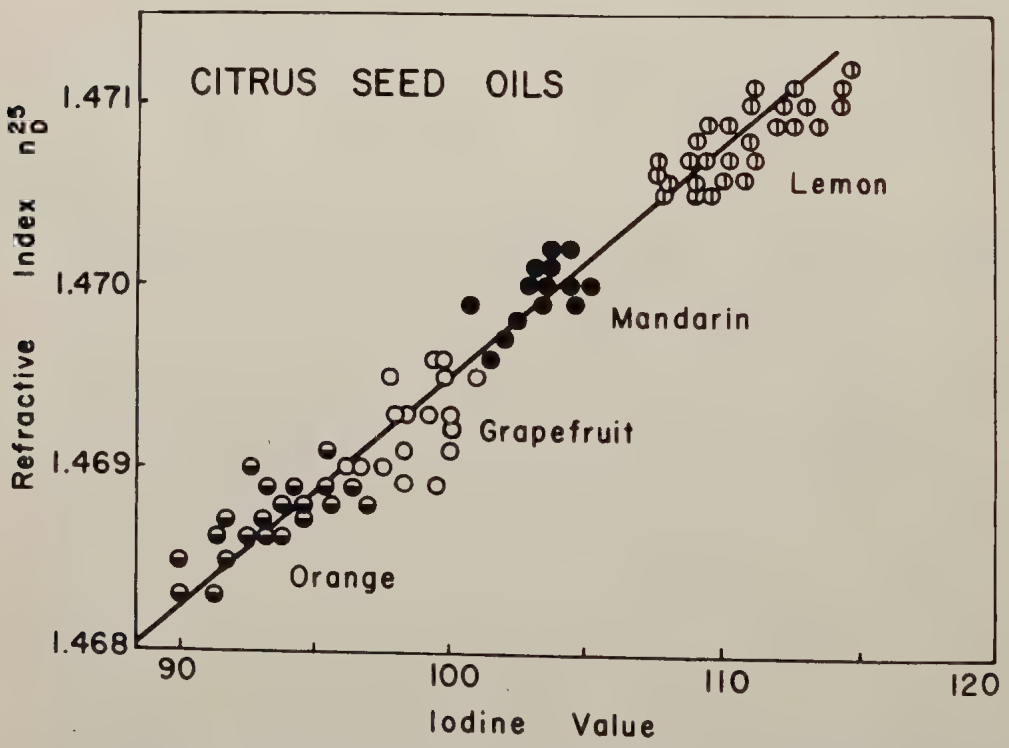


Fig. 66. A scatter diagram of refractive index measurements versus Hanus iodine absorption numbers for numerous citrus-seed oil samples. (From Hendrickson and Kesterson, 1961.)

linoleic, and linolenic acids, had unsaturated characteristics. The seed oil of the shaddock contained the same 5 fatty acids as the grapefruit-seed oil, with the exception that palmitic acid was present in lower and oleic acid in higher concentrations.

Hendrickson and Kesterson (1961) also noted the high positive correlation between the refractive indices of citrus-seed oils and their corresponding iodine absorption numbers (figure 66). The refractive index is a characteristic of each particular substance, and its measurement is a matter of considerable practical importance in determining the composition and purity of a given compound. Consequently, the increases in the refractive indices were associated with the increases in the iodine numbers of the seed oils. Since the iodine number is a measure of unsaturation in the molecule, it is also indicative of the number of double bonds in the molecule, which is a constitutive property of oil compounds.

Seed Proteins

THE MAJOR FRACTION of the proteins in seeds is considered to be composed of storage or reserve proteins. The seed proteins are usually separated and classified on the basis of solubility, but any classification based solely on physicochemical properties is not entirely satisfactory. It therefore appears that the differences in solubility may be attributed to the physical and chemical heterogeneity of mixed protein molecules. Seed proteins that have been fractionated on the basis of solubility require purification by advanced techniques in order to obtain monodispersed species.

The proximate analysis of Spanish Valencia orange seeds shown in table 102 reveals that the seeds were composed of 36.10 per cent lipid (oil), 13.75 per cent protein, and 40.78 per cent total carbohydrate. The remainder of the dry weight was made up of ash and moisture (Viguera Lobo *et al.*, 1950).

TABLE 102

PROXIMATE COMPOSITION OF SPANISH VALENCIA ORANGE SEEDS.*

Fractions	Amounts (per cent)
Moisture	6.07
Ash	3.30
Protein	13.75
Lipid (oil)	36.10
Carbohydrate, total†	40.78
(Cellulose, 8.75 per cent)	
Total	100.00

*From Viguera Lobo *et al.* (1950).
†By difference. The nitrogen-free extract was 39.48 per cent.

It is noteworthy that the concentrations of total carbohydrates and lipids of the seeds were several times the protein content.

Saunders (1931) extracted and partially purified a protein fraction from orange-seed meal. He found that the alkali halide salts in normal concentrations all extracted the same amount of nitrogen (average value, 73.4 per cent of total nitrogen) from orange-seed meal. No evidence was found for the existence of a lyotropic series in the extracting power of different salts on orange-seed meal. The protein isolated from orange-seed meal by extraction with different salts was probably the same in every case. The protein fraction had the physical and chemical properties of a globulin.

Rotha and Saunders (1932) studied the solubility properties of this protein fraction in various salt solutions. Their experimental data showed that not only were the alkali halides alike in their ability to extract the protein, but that this property was characteristic of many other salts as well. The protein fraction or globulin was found to be uniformly soluble in salts of a strong base and in weak acid, as well as in neutral salts. Globulins have been defined in terms of their solubility in salts of strong bases and in strong acids. The uniformity of the results obtained leads to the conclusion that a definite globulin was being extracted from orange-seed meal.

Bass and Saunders (1933) isolated and compared protein fractions from orange, grapefruit, and lemon seeds. The nitrogen distributions of these seed proteins are reported in table 103. They concluded from the chemical and physical properties of these proteins that they were alike in both crystalline form and nitrogen distribution. Constitutively, the seeds from the three species studied contained approximately the same percentages of total nitrogen.

Carpenter and Lovelace (1933) isolated a globulin from fat-free seeds of California Valencia oranges. This protein had an isoelectric point at pH 5.23.

TABLE 103

NITROGEN DISTRIBUTION IN PROTEINS ISOLATED FROM CITRUS SEEDS.*

Fractions	Distribution of nitrogen (in percentages of total nitrogen)		
	In orange seeds (16.29 per cent N)	In grapefruit seeds (16.21 per cent N)	In lemon seeds (16.44 per cent N)
Amino nitrogen	72.59	78.71	77.08
Nonamino nitrogen	.....	5.54	6.04
Amide and humin nitrogen	14.95	15.76	15.58
Monoamino nitrogen	52.58	51.84	52.31
Diamino nitrogen	12.14	13.20	12.48

*From Bass and Saunders (1933).

In their studies on protein solubility, O'Hara and Saunders (1937) compared the solubility of orange-seed protein with other types of seed proteins extracted with various normalities of sodium chloride (table 104). The data show that sodium chloride solutions greater than 1 *N* had little effect upon the solubility of the proteins of the seed meals of flax, orange, and peanut. In these particular meals, the material which was extracted appeared to have the characteristics of a globulin. In rye flour, the proteins in which gliadin predominated showed a type of solubility curve that was entirely different from those of the other 3 seeds in which globulin predominated.

Vitamins and Minerals in Peel and Seeds

PROVITAMIN A (CAROTENE) tests made by Fraps and Treichler (1933) showed 5 "rat units" of vitamin A per gram of dried citrus peel and pulp.[3]

Miller and Jablonski (1949) determined the relative carotene values for ungerminated and germinated grapefruit-seed embryos. The values presented here represent their averages of 15 determinations, 50 seed embryos in each lot, as milligrams per 100 grams each of fresh and dried embryo tissues.

	Fresh	*Dried*
Ungerminated embryos	0.027	0.047
Germinated embryos	0.168	0.462

After germination, when the root radicle had become 1 to 2 inches (2.5 to 5 cm) long, and the epicotyl up to 1 inch long, the increase in carotene was 6-fold on a fresh-weight basis and 10-fold on a dry-weight basis. These authors then determined the carotene values for whole, ungerminated and germinated grapefruit seeds, expressed as in the preceding tabulation.

Ungerminated seeds	0.013	0.020
Germinated seeds	0.106	0.273

Miller and Jablonski estimated that a processing factory could obtain 0.263 gram of carotene from 1 ton (908 kilograms) of ungerminated seeds and 1.59 gram from a similar amount of germinated seeds. Assuming 90 per cent of the total carotene to be β-carotene, 1 ton of germinated grapefruit seeds would contain about 2.4 million international units of provitamin A.

As noted in the discussion of pigments at the close of this chapter, grapefruit peel appears to contain little carotene after it turns yellow.

Ascorbic Acid (Vitamin C). Melas-Joannides (1939), in Greece, reported that the flavedo contained more vitamin C than the albedo. Smith *et al.* (1944) stated that the peel of size 54 (per box) grapefruit contained 142 milligrams of ascorbic acid per 100 grams. This was a relatively large amount, but not

[3] One "rat unit" is about the same as 1 international unit (IU); i.e., 0.001 milligram, determined by testing on young rats that have been reduced to a state of vitamin A deficiency.

TABLE 104

EFFECTS OF CONCENTRATION UPON THE AMOUNTS OF NITROGENOUS SUBSTANCES EXTRACTED FROM VARIOUS SEED MEALS BY SODIUM CHLORIDE.*

Normality of NaCl solution	Total N extracted from seed meals (per cent)			
	Flax (3-gram samples)	Orange (3-gram samples)	Peanut (1.5-gram samples)	Rye (5-gram samples)
Distilled water	24.27	32.24	52.86	14.81
0.125	41.73	35.59	71.15	20.32
.25	58.63	51.10	91.76	32.45
375	64.59	58.12	94.85	34.75
.50	64.50	65.54	94.55	34.13
.75	64.48	70.22	93.88	26.69
1.0	64.59	73.34	94.42	25.00
2.0	65.19	72.38	95.37	17.03
3.0	63.75	71.59	94.14	18.57
4.0	61.71	67.62	95.89	14.49
5.0	56.29	67.82	92.26	11.23
6.0	50.12	65.84	87.68	7.65
Saturated	45.53	64.92	81.23	7.65

*From O'Hara and Saunders (1937).

so great as was found in orange peel, especially Valencia. Some further data from Miller and Jablonski (1949) on the ascorbic acid content of grapefruit seeds are included in table 50 of chapter 2.

Thiamin (Vitamin B_1) and Riboflavin (Vitamin G). Roehm (1935) reported that the raw peel of grapefruit grown on the University farm in Arizona contained 0.28 IU of thiamin (vitamin B_1) and 1 IU of riboflavin (vitamin G) per gram.

Minerals. In an analysis of the peel and rag of fresh California Marsh grapefruit that had been ground together, Poore (1934) obtained 0.70 per cent ash (see table 87 of this chapter). In tests of Florida Marsh grapefruit that had been sterilized and stored, peel alone showed 0.74 per cent and rag alone showed 0.75 per cent ash on a fresh-weight basis.

Hardy and Rodriguez (1935) reported that the ash in the peel of Trinidad grapefruit of 2 different qualities and grown on 2 types of land composed from 3.7 to 4.2 per cent of the peel on a dry-weight basis (see table 58 of chapter 2).

The peel of lime-land fruit was noted as much less rich in nitrogen and potash, slightly richer in phosphate, and much richer in lime than that of acid-land fruit.

The inorganic constituents of grapefruit peel are reported with similar values for the pulp in the section headed "Minerals" in chapter 2, starting on page 118.

Pectic Substances in Peel and Rag

THE COMPOSITION AND QUANTITY of pectic constituents of grapefruit have been studied, in more or less detail, by many investigators. In the grapefruit, pectic material does not exist as a pure substance, but as complex carbohydrate derivatives of which the principal components are polyuronides composed of anhydrogalacturonic acid residues. The carboxyl groups of the polygalacturonides of the pectic constituents exist in the tissues in the free state, or combined as the esters with methyl alcohol or with the available cations. In grapefruit tissues, some of the carboxyl groups are combined with the calcium ion to form calcium pectate. Their physical and chemical properties will be considered in relation to the various pectic constituents that have been isolated from grapefruit by numerous workers.

Nomenclature. It should be borne in mind that in the earlier literature on pectic materials, many confusing terms were used that have since become obsolete, and are no longer applicable to our present chemical knowledge. As a result of this state of affairs, a committee was appointed in 1941 to revise the nomenclature of pectic substances (Kertesz *et al.*, 1944). Their definitions of the various pectic substances that occur in plant material are quoted here.

REVISED NOMENCLATURE FOR PECTIC SUBSTANCES

Pectic Substances. "Pectic substances" is a group designation for those complex colloidal carbohydrate derivatives which occur in or are prepared from plants, and contain a large proportion of anhydrogalacturonic acid units which are thought to exist in a chainlike combination. The carboxyl groups of polygalacturonic acids may be partly esterified by methyl groups and partly or completely neutralized by one or more bases.

Protopectin. The term "protopectin" is applied to the water-insoluble parent pectic substance which occurs in plants, and which, upon restricted hydrolysis, yields pectin or pectinic acids.

Pectinic Acids. The term "pectinic acids" is used for colloidal polygalacturonic acids containing more than a negligible proportion of methyl ester groups. Pectinic acids, under suitable conditions, are capable of forming gels with sugar and acid or, if suitably low in methoxyl content, with certain metallic ions. The salts of pectinic acids are either normal or acid pectinates.

Pectin. The general term "pectin" (pectins) designates those water-soluble pectinic acids of varying methyl ester content and degree of neutralization which are capable of forming gels with sugar and acid under suitable conditions.

Pectic Acids. The term "pectic acids" is applied to pectic substances mostly composed of colloidal polygalacturonic acids and essentially free from methyl ester groups. The salts of pectic acids are either normal or acid pectates.

These revised definitions were graphically illustrated by Joseph (1955) in terms of methoxyl percentage (figure 67).

The National Formulary Committee selected 7 per cent methoxyl (on a moisture-ash-free basis) as the lower limit for "pectin N.F." (Powers and

Beeler, 1940). Although not officially specified in the revised nomenclature, the 7 per cent methoxyl value makes a logical upper limit for the low-methoxyl pectins which are becoming important in the food industry.

The maximum possible methoxyl content for a completely esterified polygalacturonic acid was shown to be 16.32 per cent by Joseph *et al.* (1949). This upper theoretical limit of 16.32 per cent may be reduced to the upper practical limit of 12 to 14 per cent, as indicated by the ordinate in figure 67, by small amounts of natural carbohydrate-like materials commonly found associated with pectins, even those made in the laboratory with the greatest of care. It is highly desirable that methoxyl content be expressed as a percentage of polygalacturonic acid present, so that the presence of natural or added diluting materials in the pectin may be taken into account. It is only on such a basis of calculation that the 16.32 per cent should be considered as 100 per cent esterification.

Amounts Reported. Zoller (1918*a*) extracted the water-soluble pectic materials from grapefruit peel. From 1,560 grams of peel of 10 Florida grapefruit, he obtained 65 grams of pectic substances. A similar sample of the same variety obtained 2 months later yielded 40 grams of pectic material from 1,450 grams of peel. He reported no analytical data on this crude preparation.

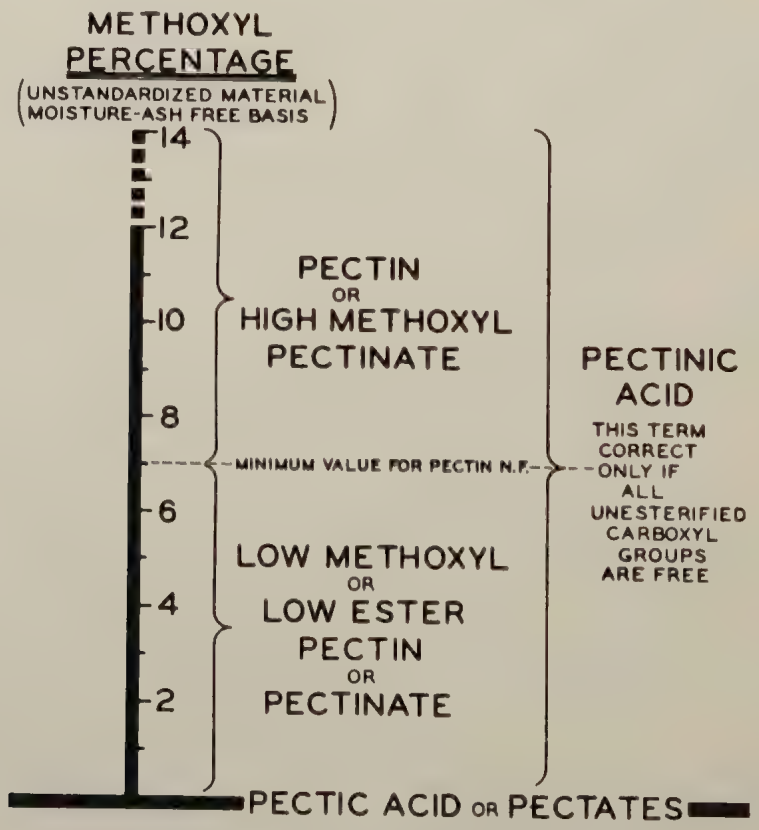


Fig. 67. Nomenclature and methoxyl content of pectic substances. (From Joseph, 1955.)

Poore (1934) found that the combined sample of peel and rag of California Marsh grapefruit contained from 3.3 to 4.5 per cent (average 3.93 per cent) pectic substances, determined as calcium pectate, on a fresh-weight basis. In another sample, the peel and rag combined had pectic material amounting to 17.85 per cent of its dry weight. Two samples of peel alone contained 3.17 and 3.64 per cent, respectively, on a wet-weight basis. In grapefruit samples from Florida, the total pectic substances, as calcium pectate, were 3.19 per cent in the peel and 3.56 per cent in the rag (see table 87, cited earlier in this chapter). On converting these values to the dry-weight basis, the peel and rag had 19.09 and 22.81 per cent total pectic substances, respectively.

Rygg and Harvey (1938) determined the changes of pectic substances in Marsh grapefruit peel as influenced by the season and by storage temperatures of 38, 46, 56, and 68° F. (3.3, 7.8, 13.3 and 20° C.), as shown in table 105. They found that, in general, the soluble pectic materials increased in the albedo during the season, and that the albedo contained more soluble pectic substances than the flavedo, with the total being significantly higher in the albedo. The data indicate that the totals in both flavedo and albedo increased during the storage period. Apparently, the moisture contents of the samples did not vary enough to produce sufficient changes in dry weight to cause this increase. The total pectic constituents were determined as calcium pectate, but no criteria of their purity were reported. It is just possible that some substance could have been produced in the peel during storage that was soluble in the extractants and was subsequently precipitated as calcium pectate.

In an investigation of Philippine fruits, Rongo and Quaitson (1940) reported on the total pectic substances of several varieties of grapefruit (*Citrus paradisi*) and pummelo (*C. grandis*). That these grapefruit varieties and the closely related species were chemically different was demonstrated by the fact that their albedo contents ranged from 16.42 to 59.76 per cent of the over-all fruit weight (table 106). The total pectic substances were determined by extracting the dried and ground albedo with 0.15 per cent hydrochloric acid, and subsequently purifying it by repeated precipitation with 95 per cent ethyl alcohol. The yield of total pectic substances varied from 16.40 to 24.98 per cent of the albedo on the dry-weight basis.

The nonpectic material in the total pectic substances determined was made evident by the yield of pectic acid obtained from the extracted pectic constituents. In other words, the pectic acid contents of the various preparations ranged from 49.95 to 68.42 per cent of the materials precipitated with 95 per cent ethyl alcohol. It is therefore apparent that, in addition to the pectic substances, the ethyl alcohol precipitated considerable quantities of extraneous materials from the dilute acid extracts of the albedo portions. On the dry weight of the albedo, the pectic acid content ranged from 8.86 to 14.89 per cent.

Methods for High Yield and Quality. Sinclair and Crandall (1949*a*) determined the pectic substances of grapefruit peel and studied their relationships to the other carbohydrate portions of the peel. The problem in this study was to adapt methods in order to obtain high yields of calcium pectate with minimum impurities. Whole peel of grapefruit was extracted with 80 per cent ethyl alcohol and separated into 2 distinct fractions, the alcohol-soluble and alcohol-insoluble solids (table 107). The carbohydrate constit-

TABLE 105

SEASONAL VARIATIONS IN THE PECTIC CONSTITUENTS OF THE PEEL OF GRAPEFRUIT.*

Dates of picking, and lot numbers	Storage temperatures (°F and °C)	Moisture content (per cent)		Pectic substances (grams of calcium pectate per 100 grams of dry peel)					
				In flavedo			In albedo		
		Flavedo	Albedo	Soluble	Protopectin	Totals	Soluble	Protopectin	Totals
OASIS, CALIFORNIA									
Oct. 29, 1935									
No. 1 (ck)..	.. ....	74.3	72.3	1.5	12.7	14.2	2.5	13.9	16.4
No. 6.....	56 (13.3)	74.1	74.1	1.6	13.7	15.3	2.9	17.8	20.7
No. 7.....	46 (7.8)	72.0	74.1	1.0	12.0	13.0	2.3	17.2	19.5
No. 8.....	38 (3.3)	72.3	73.5	1.0	13.7	14.7	2.3	16.5	18.8
Mean.....		73.2	73.5	1.3	13.0	14.3	2.5	16.4	18.9
Dec. 10, 1935									
No. 2 (ck)..	.. ....	75.0	75.5	1.6	12.6	14.2	3.1	12.6	15.7
No. 9.....	56 (13.3)	77.9	75.7	1.8	14.3	16.1	2.7	14.0	16.7
No. 10.....	46 (7.8)	74.2	76.3	1.0	11.5	12.5	2.8	14.8	17.6
No. 11.....	38 (3.3)	74.7	76.0	1.3	13.6	14.9	3.7	13.3	17.0
Mean.....		75.4	75.9	1.4	13.0	14.4	3.1	13.7	16.8
Jan. 21, 1936									
No. 3 (ck)..	.. ....	75.6	75.0	1.9	11.3	13.2	3.5	11.0	14.5
No. 12.....	56 (13.3)	77.9	78.2	1.1	11.3	12.4	3.9	14.2	18.1
No. 13.....	46 (7.8)	77.6	77.7	1.2	12.6	13.8	3.7	14.1	17.8
No. 14.....	38 (3.3)	77.6	77.8	1.3	11.2	12.5	4.4	12.4	16.8
Mean.....		77.2	77.2	1.4	11.6	13.0	3.9	12.9	16.8
Mar. 4, 1936									
No. 4 (ck)..	.. ....	78.8	81.3	1.7	12.5	14.2	5.7	11.3	17.0
No. 15.....	56 (13.3)	80.5	80.2	2.0	12.9	14.9	5.2	12.7	17.9
No. 16.....	46 (7.8)	79.1	81.2	1.7	12.5	14.2	5.4	10.7	16.1
No. 17.....	38 (3.3)	80.7	80.9	2.0	13.5	15.5	6.3	9.5	15.8
Mean.....		79.8	80.9	1.8	12.8	14.6	5.6	11.0	16.6
Apr. 14, 1936									
No. 5 (ck)..	.. ....	81.0	80.6	1.7	11.9	13.6	6.1	11.6	17.7
No. 18.....	56 (13.3)	79.1	80.7	1.1	11.4	12.5	5.3	12.7	18.0
No. 19.....	46 (7.8)	78.0	80.3	1.7	12.4	14.1	3.2	14.8	18.0
No. 20.....	38 (3.3)	77.7	80.6	1.5	14.0	15.5	4.5	13.4	17.9
Mean.....		79.0	80.6	1.5	12.4	13.9	4.8	13.1	17.9

*Adapted from Rygg and Harvey (1938).

TABLE 105—*Continued*

SEASONAL VARIATIONS IN THE PECTIC CONSTITUENTS OF THE PEEL OF GRAPEFRUIT.

Dates of picking, and lot numbers	Storage temperatures (°F and °C)	Moisture content (per cent)		Pectic substances (grams of calcium pectate per 100 grams of dry peel)					
				In flavedo			In albedo		
		Flavedo	Albedo	Soluble	Protopectin	Totals	Soluble	Protopectin	Totals
CORONA, CALIFORNIA									
Mar. 10, 1936									
No. 1 (ck)..	.. ....	77.0	78.2	1.4	10.6	12.0	6.0	9.8	15.8
No. 7.....	68 (20)	79.4	79.3	1.8	12.5	14.3	7.3	11.1	18.4
No. 8.....	56 (13.3)	78.3	78.2	1.6	13.2	14.8	6.6	11.0	17.6
No. 9.....	46 (7.8)	78.9	79.2	1.5	12.6	14.1	6.2	11.3	17.5
No. 10.....	38 (3.3)	79.7	80.0	1.5	12.4	13.9	6.8	10.1	16.9
Mean.....		78.7	79.0	1.6	12.3	13.9	6.6	10.7	17.3
April 20, 1936									
No. 2 (ck)..	.. ....	77.3	80.6	1.4	9.9	11.3	5.6	10.4	16.0
No. 11.....	68 (20)	....	80.5	...	....	....	...	....	....
No. 12.....	56 (13.3)	....	79.8	...	....	....	...	....	....
No. 13.....	46 (7.8)	....	80.0	...	....	....	...	....	....
No. 14.....	38 (3.3)	....	80.1	...	....	....	...	....	....
Mean.....		....	80.2	...	....	....	...	....	....
June 4, 1936									
No. 3 (ck)	.. ....	79.4	80.1	1.5	10.5	12.0	5.9	8.6	14.5
No. 15.....	68 (20)	81.6	81.8	2.6	13.1	15.7	8.1	9.9	18.0
No. 16.....	56 (13.3)	77.5	81.4	1.8	10.2	12.0	6.7	10.6	17.3
No. 17.....	46 (7.8)	80.3	79.4	1.8	13.3	15.1	6.9	9.3	16.2
No. 18.....	38 (3.3)	81.0	82.4	1.6	14.7	16.3	6.2	10.7	16.9
Mean.....		80.0	80.1	1.9	12.4	14.3	6.8	9.6	16.4
July 13, 1936									
No. 4 (ck)..	.. ....	82.0	81.0	1.6	11.6	13.2	6.3	10.7	17.0
No. 19.....	68 (20)	81.5	81.0	2.3	14.8	17.1	6.8	11.3	18.1
No. 20.....	56 (13.3)	79.7	80.8	2.0	12.8	14.8	6.1	11.0	17.1
No. 21.....	46 (7.8)	80.4	82.1	2.1	14.1	16.2	3.8	12.8	16.6
No. 22.....	38 (3.3)	81.2	80.7	1.8	15.4	17.2	4.8	11.7	16.5
Mean.....		81.0	81.0	2.0	13.7	15.7	5.6	11.5	17.1
Aug. 24, 1936									
No. 5 (ck)..	.. ....	79.8	81.1	1.3	12.8	14.1	5.7	11.7	17.4
No. 23.....	68 (20)	81.8	80.8	2.9	14.5	17.4	6.3	12.6	18.9
No. 24.....	56 (13.3)	77.0	79.6	2.2	10.5	12.7	6.4	13.4	19.8
No. 25.....	46 (7.8)	79.8	80.3	1.4	12.9	14.3	3.9	13.9	17.8
No. 26.....	38 (3.3)	79.8	80.4	1.6	14.3	15.9	5.3	11.4	16.7
Mean.....		79.6	80.4	1.9	13.0	14.9	5.5	12.6	18.1
Oct. 6, 1936									
No. 6 (ck)..	.. ....	81.7	80.7	2.5	14.3	16.8	7.4	11.6	19.0
No. 27.....	68 (20)	81.5	82.3	1.3	11.9	13.2	7.8	10.2	18.0
No. 28.....	56 (13.3)	81.3	80.4	2.0	11.8	13.8	5.7	12.6	18.3
No. 29.....	46 (7.8)	82.1	81.1	2.0	12.6	14.6	5.1	13.4	18.5
No. 30.....	38 (3.3)	76.6	76.8	1.1	9.1	10.2	6.8	13.9	20.7
Mean.....		80.6	80.3	1.8	11.9	13.7	6.6	12.3	18.9

uents of these 2 fractions were investigated, and some of their physical and chemical properties were determined. All analyses were made on fruit samples obtained from trees that had been fumigated with hydrocyanic acid or sprayed with oil for pest control during the growing season.

The alcohol-insoluble solids, composed chiefly of the structural constituents of the cells (cellulose, hemicellulose, pectin, protein, etc.), amounted to averages of 33.69 and 32.65 per cent of the dry weight of the peel of fumigated and oil-sprayed fruit, respectively. This fraction was well suited for a study of these structural constituents because it was free of all soluble carbohydrates and pigments, as well as of a considerable portion of the ash.

The efficiency with which alcohol separates the pectic substances of grapefruit peel is also shown in table 107. The carbon dioxide produced by the peel on hydrolysis with 12 per cent hydrochloric acid is quantitatively related to the amount of uronic acid in the sample, noted by Lefèvre and Tollens (1907). The uronic acids yielded 22.50 per cent carbon dioxide. Since pectic substances are composed chiefly of uronic acids, the yield of carbon dioxide by the fractions should give a fair approximation of the pectic content. Pectin is relatively insoluble in alcohol; therefore, most of the carbon di-

TABLE 106

PERCENTAGE YIELD OF PECTIC SUBSTANCES BY PRECIPITATION WITH 95 PER CENT ETHYL ALCOHOL.*
(From tests made in the Philippines.)

Fruits tested	Albedo content (per cent of whole fruit)	Moisture content (per cent of fresh weight)	Pectin content (per cent of dry weight)	Yield of pectic acid from extracted pectin (per cent)	Pectic acid (per cent of dry weight)
Chinese pummelo (*Citrus grandis*)	46.67	81.43	24.66	60.09	14.82
Marsh Seedless grapefruit (*C. paradisi*)	16.42	81.65	16.40	58.31	9.56
McCarty grapefruit (*C. paradisi*)	20.42	73.53	19.64	68.42	13.44
Pink pummelo (*C. grandis*)	48.84	80.77	17.73	49.95	8.86
Native pummelo (*Suha*) (*C. grandis*)	59.76	78.23	23.33	63.19	14.74
Saigon pummelo (*C. grandis*)	39.27	83.75	24.98	59.60	14.89
Siamese pummelo (*C. grandis*)	18.28	77.77	16.77	62.80	10.53
Triumph grapefruit (*C. paradisi*)	33.21	85.55	21.93	66.40	14.56

*Adapted from Rongo and Quaitson (1940).

TABLE 107

Analyses of Alcohol-insoluble and Alcohol-soluble Solids of Grapefruit Peel.*

Sample numbers	Moisture (per cent of fresh weight)	Alcohol-insoluble solids (per cent of dry weight)	Alcohol-soluble solids (per cent of dry weight)	CO_2 produced on hydrolysis with 12 per cent HCl				Per cent of methoxyl in A.I.S.†	Ratios of methoxyl to CO_2 in A.I.S.	Water-soluble sugars (as glucose) in alcoholic extract (per cent of dry weight)			Total sugars (per cent of A.S.S.)
				Alcohol-insoluble solids		Alcohol-soluble solids							
				Per cent of dry weight	Per cent of A.I.S.†	Per cent of dry weight	Per cent of A.S.S.			Reducing	Sucrose	Totals	
Fumigated Fruit													
1.....	81.46	33.47	66.53	3.51	10.48	0.42	0.63	5.71	0.545	24.12	15.89	40.01	60.14
2.....	81.33	33.42	66.58	3.46	10.35	.45	.68	5.66	.547	24.08	15.25	39.33	59.07
3.....	81.52	33.69	66.31	3.53	10.48	.44	.66	5.73	.547	23.97	16.68	40.65	61.30
4.....	81.40	33.84	66.16	3.54	10.47	.42	.63	5.78	.552	24.19	16.04	40.23	60.81
5.....	81.46	34.04	65.96	3.55	10.43	.49	.74	5.71	.547	25.38	15.84	41.22	62.49
Means...	81.43	33.69	66.31	3.52	10.44	.44	.67	5.72	.548	24.35	15.94	40.29	60.76
Oil-sprayed Fruit													
1.....	82.23	32.95	67.05	3.45	10.48	0.49	0.73	5.71	0.545	24.87	17.46	42.33	63.13
2.....	82.01	32.65	67.35	3.37	10.32	.39	.58	5.66	.548	24.53	15.57	40.10	59.55
3.....	82.13	32.73	67.27	3.41	10.42	.51	.76	5.75	.552	25.98	16.34	42.32	62.90
4.....	82.11	32.26	67.74	3.34	10.37	.39	.58	5.78	.557	25.31	16.24	41.55	61.34
5.....	82.23	32.65	67.35	3.37	10.32	.33	.49	5.66	.548	24.91	15.17	40.08	59.50
Means...	82.14	32.65	67.35	3.39	10.38	.42	.63	5.71	.550	25.12	16.16	41.28	61.28

*From Sinclair and Crandall (1949*a*).
†Calculated on an ash-free, moisture-free basis.

oxide should be liberated in the alcohol-insoluble solids fraction. This fraction yielded, on the average, 10.44 and 10.38 per cent of its weight as carbon dioxide in fumigated and oil-sprayed fruit, respectively.

The high percentage of carbon dioxide in the alcohol-insoluble solids resulted from the fact that a large proportion of the dry matter was soluble in 80 per cent alcohol. Since 80 per cent alcohol does not remove pectin from the peel, it follows that the lower the percentage of alcohol-insoluble solids in the dry matter, the higher the percentage of carbon dioxide in this fraction. On the assumption that all the carbon dioxide liberated from the alcohol-insoluble fraction was derived from the pectin, the percentage of pectin in this fraction can be estimated by the simple calculation $10.44 \times 100/17.40$, where 17.40 is the amount of carbon dioxide determined on highly purified calcium pectate.

The alcohol-insoluble solids in the peel of fumigated and oil-sprayed fruit contained, on the average, 5.72 and 5.71 per cent methoxyl (table 107). Since these values are easily determined by saponification with sodium hydroxide, a quick method is available for the approximate determination of pectin. The uronic acid carboxyl groups of pectin are free for salt formation or esterification, but the hemicelluloses do not exhibit acidic properties when treated with alkali at room temperatures. The pectin content of a plant tissue, calculated from the methoxyl values, depends on the methoxyl content of pure pectin. Since the methoxyl content of pectin varies with the source, method of preparation, and other variables, an experimental factor must be determined from a pure pectin prepared by a definite procedure. As mentioned earlier, the methoxyl content of a completely esterified polygalacturonide is theoretically 16.32 per cent, but the use of this figure for calculating a conversion factor would be impractical, since pectins containing such a high concentration of methoxyl have not been isolated as yet.

The ratio of methoxyl to carbon dioxide liberated is much lower in grapefruit peel than in lemon peel. In comparison with the carbon dioxide values of the alcohol-insoluble solids, the methoxyl content of these fractions should be higher than those reported (table 107). This is substantiated by the fact that the pectin equivalent of the carbon dioxide is greater than that equivalent to the methoxyl content, on the assumption that pectin contains, on the average, 11 per cent methoxyl. The relatively low methoxyl content and the absence of free acidity indicate that some of the carboxyl groups are combined with the cations of the alcohol-insoluble fraction. This can be shown experimentally by converting the methoxyl values to equivalent percentages of carbon dioxide plus the carbon dioxide calculated from the free acidity of the ash-free alcohol-insoluble solids. For example, the carbon dioxide that is equivalent to 5.72 per cent methoxyl (average value) is 8.12 per cent; the carbon dioxide that is equivalent to the free acidity (23.4 milligrams of

sodium hydroxide per gram) of the ash-free alcohol-insoluble solids is 2.57 per cent; the sum of 8.12 + 2.57 = 10.69 per cent total carbon dioxide. This value is in good agreement with 10.44 per cent carbon dioxide, which was the amount determined on the alcohol-insoluble solids by hydrolysis with 12 per cent hydrochloric acid.

A comment should be made on the determination of the combined carboxyl groups in the alcohol-insoluble solids. In this fraction, the nonesterified carboxyl groups occur in the pectin and are combined with the mineral constituents. The mineral bases are freed from the alcohol-insoluble solids by extracting the latter with 0.1 *N* hydrochloric acid in 80 per cent alcohol. The acidulated alcohol lowers the acidity to pH 1, liberating the carboxyl groups and forming the chlorides of the minerals. After the samples have been washed free of chlorides with 80 per cent alcohol, the free carboxyl groups can be titrated with a standard sodium hydroxide solution.

The alkaline ash of the alcohol-insoluble solids averaged 0.780 milliequivalents per gram of sample. The alkaline ash is a measure of the cations combined with organic substances in the alcohol-insoluble fraction. The substances in combination with the mineral constituents are chiefly pectin and a smaller amount of organic acids that are insoluble in 80 per cent alcohol. Since the alcohol-insoluble fraction contains more than 50 per cent pectin, it is reasonable to expect the mineral constituents to be combined with the nonesterified carboxyl groups, which are equal to 0.584 milliequivalents per gram. The difference between the alkaline ash and the nonesterified groups is equal to the minerals combined with organic substances other than pectin. In equational form, 0.780 – 0.584 = 0.196 milliequivalents per gram of alcohol-insoluble solids.

In the peel of the grapefruit samples, the dry matter soluble in 80 per cent ethyl alcohol averaged 66.31 and 67.35 per cent, respectively (table 107). These results were higher than those reported for lemon peel by Sinclair and Crandall (1949*b*). They were approximately equivalent to the values determined by Harvey and Rygg (1936*c*), who found that the soluble solids and total sugars in grapefruit peel increased steadily through the season, and that the total sugars accounted for slightly more than half of the soluble solids. Swift and Veldhuis (1957) reported similar trends for the increase in soluble solids and soluble carbohydrates of the peel of Florida oranges during growth and maturation. It is fairly well established that the soluble carbohydrates in the peel show seasonal increases in all commercial varieties.

In the present experiment, the total sugars averaged 60.76 per cent and 61.28 per cent, respectively, of the alcohol-soluble solids in the peel of fumigated and oil-sprayed fruit. On a dry-weight basis, the sugars averaged 40.29 and 41.28 per cent. The amount of reducing sugars was considerably greater than that of sucrose. Fluctuations in the concentration of these peel constit-

uents were markedly influenced by the maturity of the fruit and the environmental and climatic conditions under which the fruit was grown. In the alcoholic extract from both sets of samples, approximately 40 per cent of the solids was in forms other than reducing sugars and sucrose. The alcohol-soluble fraction contained substances which liberated a small amount of carbon dioxide (0.67 per cent average for fumigated samples and 0.63 per cent for oil-sprayed samples) on hydrolysis with 12 per cent hydrochloric acid.

In studying the carbohydrates of grapefruit peel, Ting and Deszyck (1961) found that 80 to 90 per cent of the alcohol-soluble solids was composed of soluble sugars, and that the alcohol-soluble solids increased as the fruit matured. A similar trend was noted for the total pectic substances of the peel when the values were reported as percentages of the alcohol-insoluble solids. The separation of the colloidal and noncolloidal substances is conveniently made by extracting the peel samples with 80 per cent ethyl alcohol. This concentration of alcohol is reasonably effective in precipitating the colloids, such as pectic substances, dextrins, starches, celluloses, hemicelluloses, lignins, proteins, etc., and in dissolving the noncolloidal substances, such as soluble carbohydrates, glucosides, inorganic nitrogen, and amino acids. Note that under certain experimental conditions, overlapping solubilities between these 2 fractions can and do occur.

Sinclair and Crandall (1949*a*) noted that pectic substances were extracted from the alcohol-insoluble solids of whole peel with hot water and by hydrolysis with dilute hydrochloric acid (table 108). The values were determined as calcium pectate, reported as percentages of the alcohol-insoluble solids. The calcium pectates from the water-soluble pectin of fumigated and oil-sprayed fruit averaged 32.50 and 31.96 per cent, respectively. The calcium pectates from the acid-soluble pectin of the same samples averaged 11.29 and 11.99 per cent. The total calcium pectate extracted (water-soluble and acid-soluble) was 43.79 per cent for the fumigated fruit and 43.95 per cent for the oil-sprayed fruit.

The degree of purity of the calcium pectate values can be ascertained from their respective carbon dioxide, calcium, and furfural values. The yields of carbon dioxide from the calcium pectates were slightly higher than the 17.40 per cent mean obtained by Joseph and Bryant[4] for highly purified calcium pectates from lemon pectin. The calcium contents were higher than those usually reported for calcium from citrus (Gaddum, 1934) and from apple pectins, and they were higher than would be expected from the carbon dioxide content; i.e., on the basis that calcium pectate should contain 7.45 per cent calcium. Gaddum showed that the calcium content of calcium pectates prepared from pectins which had been repeatedly precipitated with alcohol

[4] G. H. Joseph and E. F. Bryant. Unpublished data. Sunkist Growers, Inc., Research Department, Corona, California.

TABLE 108

Water-soluble and Acid-soluble Pectin of Alcohol-insoluble Solids of Grapefruit Peel, Determined as Calcium Pectate.*

Sample numbers	Total CO_2 (per cent of A.I.S.)	Water-soluble pectin as calcium pectate				Acid-soluble pectin as calcium pectate				Total calcium pectate (per cent of A.I.S.)	Insoluble residue (per cent of A.I.S.)
		Totals (per cent of A.I.S.)	Constituents (per cent)			Totals (per cent of A.I.S.)	Constituents (per cent)				
			CO_2	Calcium	Furfural		CO_2	Calcium	Furfural		
Fumigated Fruit											
1	10.48	34.33	18.04	8.65	18.45	11.38	17.85	7.82	17.71	45.71	41.10
2	10.35	33.61	18.08	9.18	19.30	11.07	18.20	7.70	17.08	44.68	41.15
3	10.48	30.93	18.45	9.00	18.05	11.01	18.42	8.08	18.13	41.94	41.00
4	10.47	30.98	18.55	9.00	18.70	11.51	18.15	8.00	17.13	42.49	41.20
5	10.43	32.65	18.65	9.02	19.65	11.46	18.37	7.22	17.48	44.11	37.40
Means	10.44	32.50	18.35	8.97	18.83	11.29	18.20	7.76	17.51	43.79	40.37
Oil-sprayed Fruit											
1	10.48	32.60	18.72	8.91	19.01	11.90	18.07	7.33	17.42	44.50	39.80
2	10.32	31.70	18.40	8.89	19.37	12.06	18.08	7.54	18.07	43.76	40.22
3	10.42	31.60	18.39	9.02	19.00	11.92	17.63	7.77	17.70	43.52	41.21
4	10.37	31.78	18.50	9.02	19.66	11.99	18.47	7.54	18.38	43.77	41.90
5	10.32	32.15	18.41	8.98	19.62	12.07	18.15	7.37	17.33	44.22	41.26
Means	10.38	31.96	18.48	8.96	19.33	11.99	18.08	7.51	17.78	43.95	40.88

*From Sinclair and Crandall (1949*a*).

approached a constant value of 7.50 per cent. In his water-soluble albedo pectins, the initial high calcium values were associated with the presence of high ash. In the present study, the purity of calcium pectates can be shown by the fact that the alkalinity of the ash was in good agreement with the percentages of calcium.

The uniformity of the calcium content of calcium pectate is evidence that the pectin precipitated by the calcium ion is a chemical substance of considerable uniformity. The claim is frequently and justly made that the calcium pectate method determines the pectin, but very few of its decomposition products. The close association of pectin with the other cell-wall constituents is such that extraction of pectin by chemical methods would result in a calcium pectate of variable composition, especially if the extraction processes were prolonged. The calcium pectate method can be used to estimate the pectin content of citrus peel if proper care is used in extracting the pectin from the tissue. In the present study, the calcium in calcium pectate from the water-soluble pectin was higher than in the acid-extracted pectin (table 108).

The furfural contents of the various calcium pectates were in accord with values reported by Norris and Resch (1935) on pectins from other sources, and especially did they agree with the results of Bryant *et al.* (1944) on the pectin of lemon (*National Formulary,* edition VIII), which contains a high content of galacturonic acid (87.6 per cent). No claim is made that pectic acid can be freed of arabinose and galactose by precipitation as the insoluble calcium pectate. The complete removal of galactose and arabinose from pectin by hydrolysis (as by hydrochloric acid in methyl alcohol) results in a degraded form of pectic acid. On the basis of these analyses, the calcium pectate values given in table 108 represent quite accurately the pectin content of these extracts.

The residue left after extracting the pectins from alcohol-insoluble solids (table 108) was composed chiefly of cellulose and hemicellulose (Sinclair and Crandall, 1949*a*). This residue contained a comparatively small amount of firmly bound pectin that was difficult to extract and determine quantitatively. As noted in the table, the percentages of this fraction determined on the different peel samples were somewhat variable, depending upon the methods used to extract the pectin. The extractants used to dissolve the pectin also dissolved various portions of the hemicellulose. All the carbon dioxide could be removed from the insoluble residue by prolonged acid hydrolysis, but the calcium pectate determinations made on these hydrolysates were highly contaminated with hemicellulose impurities. Attempted purification of the calcium pectate by redissolving and reprecipitation did not remove all the contaminants.

All the residues that were tested contained carbon dioxide, and thus showed the presence of uronic acids (table 108). After thorough extraction of the

alcohol-insoluble solids with hot water and dilute hydrochloric acid, the residues still contained pectin or uronic acids combined with celluloses or hemicelluloses. The carbohydrate reserve was definitely low, as shown by the absence of starch and dextrin. No substantial amount of evidence exists to prove that the hemicellulose fraction acts as a carbohydrate reserve.

Sinclair and Crandall (1949*a*) also prepared a balance sheet showing the relationship of carbon dioxide in the alcohol-insoluble fraction to the amount determined in calcium pectate from aqueous and acid extracts. The degree of efficiency with which the calcium ion precipitates pectin in grapefruit peel can be observed from the data in table 109 by comparing the carbon dioxide contents of the various extracts with the corresponding carbon dioxide values of the calcium pectates. For the fumigated and oil-sprayed samples, the average amounts of carbon dioxide in the aqueous extracts were, respectively, 6.38 and 6.36 per cent of the alcohol-insoluble solids. The corresponding values for the calcium pectates prepared from these extracts were 5.96 and 5.91 per cent. In the 2 groups of samples, the amounts of carbon dioxide in the calcium pectates accounted for averages of 93.44 and 92.91 per cent of the totals in the aqueous extracts.

Experiments similar to those on the aqueous extracts were performed on the acid extracts of the alcohol-insoluble solids. In these experiments, the amounts of carbon dioxide in the calcium pectates accounted for averages of 91.72 and 93.31 per cent of the totals in the acid extracts.

The foregoing results show that more than 90 per cent of the uronic acid carbon dioxide extracted from the alcohol-insoluble solids with water and dilute acid solution was precipitated as calcium pectate. A portion of the loss in carbon dioxide undoubtedly occurred in the large volume of wash water used to free the calcium pectate of chlorides. The fact that most of the carbon dioxide occurred in the galacturonic acid of the calcium pectate indicates that only a small amount of uronide, if any, could have been combined with the hemicellulose dissolved by the water and dilute hydrochloric acid solutions in the extraction process.

Another important point is the relation of the carbon dioxide of the alcohol-insoluble solids to the sum of that of the various fractions. As shown in table 109, the sums of the carbon dioxide in the aqueous extract, acid extract, and residue averaged 9.84 and 9.90 per cent of the alcohol-insoluble solids. The latter values were equivalent to 94.26 and 95.19 per cent of the total carbon dioxide content of the alcohol-insoluble solids. Thus, approximately 5 per cent of the total was lost in the extraction processes.

Rouse (1953) reported on the distribution of the total pectic substances, determined as calcium pectate, in various component parts of citrus fruits. The data presented in table 110 compare the pectic substances of Marsh Seedless and Duncan grapefruit with those of 4 varieties of Florida oranges. The

TABLE 109

BALANCE SHEET, SHOWING RELATIONSHIP OF CARBON DIOXIDE IN ALCOHOL-INSOLUBLE SOLIDS TO AMOUNT DETERMINED IN CALCIUM PECTATE FROM AQUEOUS AND ACID EXTRACTS.*

Sample numbers	Total CO_2 (per cent of A.I.S.)	CO_2 in aqueous axtract			CO_2 in acid extract			CO_2 in residue (per cent of A.I.S.)	Sum of CO_2 fractions determined (per cent of A.I.S.)	Percentage of total CO_2 determined
		Totals (per cent of A.I.S.)	Equivalent to Ca pectate in extract (per cent of A.I.S.)	Percentage accounted for	Totals (per cent of A.I.S.)	Equivalent to Ca pectate in extract (per cent of A.I.S.)	Percentage accounted for			
FUMIGATED FRUIT										
1	10.48	6.37	6.19	97.17	2.15	2.03	94.42	1.26	9.78	93.32
2	10.35	6.37	6.08	95.45	2.29	2.01	87.77	1.26	9.92	95.85
3	10.48	6.33	5.71	90.21	2.23	2.03	91.03	1.37	9.93	94.75
4	10.47	6.32	5.75	90.98	2.26	2.09	92.48	1.34	9.92	94.75
5	10.43	6.52	6.09	93.40	2.26	2.10	92.92	0.88	9.66	92.62
Means	10.44	6.38	5.96	93.44	2.24	2.05	91.72	1.22	9.84	94.26
OIL-SPRAYED FRUIT										
1	10.48	6.67	6.10	91.45	2.30	2.15	93.48	1.19	10.16	96.95
2	10.32	6.27	5.83	92.98	2.36	2.18	92.37	1.23	9.86	95.54
3	10.42	6.22	5.81	93.41	2.30	2.10	91.30	1.29	9.81	94.15
4	10.37	6.27	5.88	93.78	2.30	2.21	96.09	1.19	9.76	94.12
Means	10.40	6.36	5.91	92.91	2.32	2.16	93.31	1.22	9.90	95.19

*From Sinclair and Crandall (1949*a*).

TABLE 110

DISTRIBUTION OF TOTAL PECTIN IN THE COMPONENT PARTS OF CITRUS FRUITS.*

Varieties of fruit	Flavedo			Albedo			Rag		
	Per cent moisture	Per cent Ca pectate (wet basis)	Per cent Ca pectate (dry basis)	Per cent moisture	Per cent Ca pectate (wet basis)	Per cent Ca pectate (dry basis)	Per cent moisture	Per cent Ca pectate (wet basis)	Per cent Ca pectate (dry basis)
Hamlin orange	76.3	4.3	18.2	75.8	4.5	18.8	83.1	2.5	14.7
Pineapple orange	73.6	3.9	14.8	72.1	4.4	15.7	78.2	4.3	19.9
Temple orange	77.5	2.4	10.8	73.9	4.4	16.7	81.3	3.1	16.5
Valencia orange	72.4	5.0	18.1	68.2	8.7	27.5	78.4	7.7	35.4
Marsh Seedless grapefruit	78.1	6.8	31.2	80.5	2.7	13.9	82.4	3.5	20.0
Duncan grapefruit	77.3	4.5	20.0	78.6	2.8	13.0	82.8	4.1	24.1

Varieties of fruit	Juice vesicles			Seeds			Juice		
	Per cent moisture	Per cent Ca pectate (wet basis)	Per cent Ca pectate (dry basis)	Per cent moisture	Per cent Ca pectate (wet basis)	Per cent Ca pectate (dry basis)	Per cent moisture	Per cent Ca pectate (wet basis)	Per cent Ca pectate (dry basis)
Hamlin orange	85.1	2.3	15.5	44.7	2.5	4.5	90.3	0.07	0.8
Pineapple orange	83.6	1.8	11.1	54.1	3.5	7.5	87.2	.11	.9
Temple orange	83.0	1.5	9.0	46.7	3.4	6.4	86.8	.05	.4
Valencia orange	84.0	2.5	15.3	54.8	3.8	8.4	88.9	.09	.8
Marsh Seedless grapefruit	87.8	1.8	14.6	57.1	2.3	5.4	91.7	.08	.9
Duncan grapefruit	87.5	1.6	12.7	54.7	2.2	5.1	90.1	.08	.8

*From Rouse (1953).

juice in all instances contained the lowest concentrations of pectic material on both the wet and dry bases. The pectic substances in the juice vesicles varied from 9 to 15.5 per cent of the dry weight. Of the 2 grapefruit varieties studied, the Marsh Seedless had the greatest amount of pectic material in the flavedo (31.2 per cent). The rag of both varieties had a much higher pectic content (dry-weight basis) than the albedo components of the peel. When the pectic contents of the various grapefruit components are considered on a fresh-weight basis, the values can be arranged in the descending order: flavedo, rag, albedo, seeds, juice vesicles, and juice.

Chemical and Physical Properties of Pectin Associated with Quality and Quantity of the Commercial Product. Pectin and protopectin are both derivatives of pectic acid, and the carboxyl groups are esterified with methoxyl groups. The structures of pectin and protopectin are composed of linkages of galacturonic acid residues into long chains, with varying degrees of esterification of the carboxyl groups. In plant cells and tissues, pectin is associated with hemicelluloses and celluloses in a water-insoluble form called "protopectin," which is the precursor of the water-soluble pectin of commerce. Protopectin can be converted into water-soluble pectin by hydrolysis with heat, acids, warm or cold alkali, and certain enzymes. A completely esterified polygalacturonic acid theoretically contains 16.32 per cent methoxyl, but with the association of hemicellulosic and cellulosic materials with the pectic substances, the maximum methoxyl content is reduced to the upper practical limit of 12 to 14 per cent. The methoxyl content of commercial pectins is usually between 8 and 11 per cent. The low-methoxyl pectins (3.5 to 5 per cent) have part of the carboxyl groups combined as methyl esters, part as salts of alkali or alkaline earth metals, and part as ammonium salts; in some instances, portions of the carboxyl groups are converted into acid amides. (Joseph, 1955; figure 67; "Revised Nomenclature," page 223.)

The chemical nature of the pectin molecules can be ascertained by taking advantage of certain chemical reactions of polygalacturonic acid which can be determined quantitatively. The free carboxyl groups can be titrated directly with standard alkali. The methoxyl content is determined by saponifying the pectin with a known concentration of standard alkali, subsequently adding an equivalent amount of standard hydrochloric acid, and then back-titrating the solution with standard sodium hydroxide. The combined ammonium and acid amide content can be determined in the distillate from a sodium hydroxide solution of the pectic material. The total galacturonide content of pectin can be calculated from the carbon dioxide produced on hydrolysis with 19 per cent hydrochloric acid. With the loss of carbon dioxide during this hydrolysis, the pentose arabinose is formed which, on further heating, is quantitatively converted into furfural by losing 3 molecules of water.

The carbon dioxide is absorbed in barium hydroxide and is determined by titration, while the furfural is determined colorimetrically by reacting with aniline. Galacturonic acid can also be determined in the hydrolytic products of pectin by the colorimetric carbazole method.

Essential Oils and Peel Wax

THE ESSENTIAL OILS are those ethereal oils in plant tissues that are volatile with steam and separate as an oily layer in the distillate. Chemically, the essential oils include terpenes, sesquiterpenes, di- and higher terpenes, many derivatives of these substances, and nonterpene compounds. The nonterpene compounds include many organic structures, such as mercaptans, anthranilic acid esters, alcohols, esters, and various hydrocarbons. The physical and chemical properties of the essential oils are entirely different from those substances that are classed as natural fats, oils, and related substances.

For a detailed discussion of the essential oils of orange fruits, see Kirchner (1961). His review covered the literature through 1960, and included not only the constituents of the essential oils of the orange fruit, but also the production methods employed and the factors affecting the yield and stability of the peel oils. Consequently, the information reported in this section is restricted chiefly to the essential oils of grapefruit, except in instances where related data on other citrus species are used to enhance the discussion.

Because of their flavoring qualities, citrus essential oils have been used in a wide variety of products. Some of the industries in which they are employed are: perfumery, toilet goods, beverages, extracts, bakery goods, canning, condiments, confectionery, food industry (general), ice cream, preserves, pharmaceuticals, and soap.

Hendrickson and Kesterson (1951) pointed out that the so-called essential oils are not true oils, chemically speaking. They fall into 2 groups that are quite distinct chemically. One of these consists of the terpenes, which are hydrocarbons of the general formula $C_{10}H_{16}$ of which a large number of isomers exist. The essential oils of the second group all contain oxygen and some sulfur. They are complex alcohols, aldehydes, and ketones, many of them being phenol derivatives. The oxygenated compounds, the principal flavoring agents of citrus oils, may constitute from 1.5 to 6.5 per cent of the extracted oil. Cold-pressed peel oil also contains some waxes from the cuticle, which are dissolved in the essential oils during pressing; see page 267.

Citrus essential oils are contained in sacs or vesicles in the flavedo, the outer, colored portion of the peel. The cells surrounding the vesicles contain an aqueous solution of sugars, salts, and colloids, and exert a certain amount of pressure on the oil vesicles. For more detailed descriptions of the oil vesicles, see Winton and Winton (1935), Braverman (1949), and Bartholomew and Sinclair (1951).

Nelson and Mottern (1934) stated that Florida alone was processing 100,000 tons of grapefruit annually by 1934. They estimated that 1 pound (0.454 kilogram) of oil could be obtained from each ton (908 kilograms) of fruit. Peel oil was then new to commerce, its manufacture having begun in 1933, but it almost immediately found favor in England as a flavoring for beverages.

Methods of Preparation. The first descriptions of the composition and characteristics of grapefruit peel oil were given by Zoller (1918*b*). His results are said to be open to criticism because he used such a small amount of oil: 100 grams, steam-distilled.

Nelson and Mottern (1934) used 16 liters (almost 17 quarts) of pressed (Pipkin-extracted) grapefruit oil. They described the steps necessary at that time for making terpeneless oil. Such oil can readily be made to form a concentrated, stable emulsion that is satisfactory to many commercial users as a flavoring medium. It is free of the turpentine taste, which is caused by the oxidation products of limonene.

Kirchner and Miller (1952) devised a chromatographic method for the preparation of terpeneless essential oils. They stated that the terpeneless oils produced by chromatography in each case had a delicate odor, showing much improvement over the original oil. In comparison, none of the commercial terpeneless oils examined had the same odor quality. These commercially produced terpeneless oils, when examined by the chromatostrip method, showed considerable quantities of hydrocarbon fraction still present.

Components, Physical Characteristics, and Amounts. Zoller (1918*b*) made determinations on distilled oil, for which he reported these properties and physical characteristics: d_{20}° (specific gravity at 20° C. or 68° F.), 0.845 to 0.860; n (refractive index at 20° C.), 1.4750 to 1.4785; $[\alpha]$ (optical rotation in 100-millimeter [4-inch] tube), + 72.5 to 78.5°; D-limonene, 90 to 92 per cent; citral, 3 to 5 per cent; D-pinene, 0.5 to 1.5 per cent; linaloöl, 1 to 2 per cent. In general, this oil had characteristics that were intermediate between lemon and orange oils.

McNair (1926–27) reported the odor of grapefruit oil as being much like that of citral, and its color as clear greenish yellow. He gave the same values for refractive index, optical rotation, and specific gravity as those given above by Zoller, though he did not so state. He made tests for A-pinene, D-limonene, linaloöl, citral, and geraniol, but did not give the amounts determined.

Nelson and Mottern (1934), in their tests of pressed grapefruit oil, reported about 90 per cent limonene, 2 to 3 per cent oxygenated volatile constituents, sesquiterpenes, and 7 to 8 per cent waxy materials. Other constituents reported are given under the subheading "Aldehydes" later in this chapter. Examinations for pinene, terpineol, and decyl alcohol gave negative results, but a small quantity of nonyl alcohol may have been present. Octylic and

decylic acids were present in small quantities, probably as esters. Table 111 gives the physical characteristics of grapefruit oil reported by these workers on the basis of 7 tests.

Poore (1934), by distillation, found 0.56 per cent essential oil in the fresh peel and rag of California grapefruit. Similar tests on Florida peel yielded 0.43 per cent oil. *(See table 87, page 201.)*

Braverman and Monselise (1940) determined the amount of oil in grapefruit peel at weekly intervals between December, 1939, and March, 1940, in Palestine. The report showed values ranging from 28.20 to 43.30 milligrams per gram. They found no regular or consistent changes in the amounts of oil as the season advanced, and there was no correlation between the amount of rainfall and the amount of oil.

Dutt and Prakash (1949), in India, found grapefruit oil to have the following physical characteristics: n_D^{19} (refractive index), 1.4781; d_{19}^{0} (specific gravity), 0.8519; $[\alpha]_D^{19}$ (optical rotation) (in $CHCl_3$ [chloroform]), 111.3°; acid value, 0; saponification value, 6.3; saponification value after acetylation, 14.7. They also found: D-limonene, 92.72 per cent; methyl anthranilate, 0.64 per cent; linaloöl, 0.9 per cent; crystalline wax, 0.53 per cent; noncrystalline wax, 2.05 per cent; crystalline coumarin derivative (M 231°), 1.06 per cent; loss and unidentified, 2.1 per cent.

Feigenbaum (1949), in Palestine, reported that about 1 kilogram of oil was obtained from each ton of Marsh grapefruit. The oil was recovered from the peel by the old, classical sponge-and-hand method, or by the more recent mechanical methods. He reported the following average values for the peel oil: d_{15} (specific gravity), 0.850 to 0.859; $[\alpha]_D^{20}$ (optical rotation), 91 to 98°; n^{20} (refractive index), 1.474 to 1.477.

Guenther (1949) gave the following values (obtained by the New York laboratories of Fritzsche Brothers, Inc.) for Brazilian grapefruit oil: specific gravity at 25° C., 0.858; optical rotation at 25°, + 93° 40′; refractive index at 20°, 1.4755; aldehyde content, calculated as decyl aldehyde (hydroxylamine hydrochloride method), 1 per cent; evaporation residue, 6.9 per cent.

TABLE 111
ANALYSES OF FLORIDA GRAPEFRUIT OIL.*
(Stated as ranges and averages.)

Specific gravity d_{20}^{20}	Optical rotation $[\alpha]_D^{20}$	Refractive index n_D^{20}	Aldehyde as acetyl† and decyl (per cent)
0.8543–0.8579	92.88–93.83	1.4753–1.4760	1.60–1.74
0.8563	93.28	1.4758	1.67

*From Nelson and Mottern (1934).
†U.S.P. method for citral.

With the development of more precise physical and chromatographic methods for determining natural products in plant tissues, it is now possible to identify constituents which hitherto existed in such low concentrations that it had been impossible to identify and determine their physical and chemical properties. In the analyses of the essential oils of citrus fruits, scientists have used methods such as spectrophotometric analyses in the ultraviolet, infrared absorption, gas chromatography, and chemical analyses to obtain data which have thrown considerable light on the biochemistry and physiology of fruit metabolism.

The data reported in table 112 show the physical and chemical characteristics of cold-pressed Marsh grapefruit oil, determined on samples varying in size from small and immature to fully mature fruit (Kesterson and Hendrickson, 1963). These data indicate that certain values, such as specific gravity, refractive index, and the percentages of free alcohol and total alcohol, decreased with fruit maturity, while the values for optical rotation and aldehyde content increased, progressing from samples A through D. The percentages of evaporation residue and free acid showed a gradual tendency to diminish with fruit maturity. The ester values before and after acetylation did not change significantly during the sampling period. The total carbonyl content increased from 0.41 to 1.75 per cent as the fruit matured. When the

TABLE 112

PHYSICAL AND CHEMICAL PROPERTIES OF COLD-PRESSED MARSH GRAPEFRUIT OIL.*

Constituents and related data	Sample designations			
	A	B	C	D
Specific gravity (25°/25° C.)	0.8609	0.8595	0.8571	0.8557
Refractive index n_D^{20}	1.4791	1.4784	1.4778	1.4771
Refractive index 10 per cent distillate n_D^{20}	1.4725	1.4723	1.4719	1.4717
Difference	0.0066	0.0061	0.0059	0.0054
Optical rotation $[\alpha]_D^{25}$	+87.52	+88.32	+89.34	+90.54
Optical rotation 10 per cent distillate $[\alpha]_D^{25}$	+96.67	+96.90	+97.10	+97.30
Difference	+9.15	+8.58	+7.76	+6.79
Evaporation residue (per cent)	10.91	11.59	9.65	8.96
Aldehyde content (per cent)	0.41	0.73	1.19	1.75
Acid number	1.54	1.77	1.11	0.88
Free acid (per cent)	0.39	0.45	0.28	0.23
Ester number before acetylation	12.37	11.14	11.14	11.68
Ester content before acetylation (per cent)	4.32	3.89	3.89	4.08
Ester number after acetylation	12.65	13.27	13.98	13.00
Ester content after acetylation (per cent)	4.42	4.64	4.89	4.54
Free alcohol (per cent)	0.48	0.38	0.28	0.26
Total alcohol (per cent)	3.88	3.72	3.34	3.47

*From Kesterson and Hendrickson (1963).

carbonyl carbon is terminal and carries a hydrogen, as in this instance, the compound in which it occurs is an aldehyde; when it carries 2 carbons, the compound it represents is a ketone.

Kesterson and Hendrickson (1963) also followed the seasonal changes in the composition of terpeneless Marsh grapefruit oils by means of gas chromatographic analyses (table 113). It is well known that cold-pressed grapefruit oil is relatively high in terpenes and low in oxygenated compounds. In

TABLE 113

CHANGES IN COMPOSITION OF TERPENELESS MARSH GRAPEFRUIT OIL AS RELATED TO FRUIT MATURITY.*

(Determined by gas-liquid chromatography [GLC].)

Compounds†	Sample numbers	Sample designations			
		A	B	C	D
Air	1	x	x	x	x
α-Pinene	2	2.5	2.8	1.6	0.5
(?)	3	0.3	0.4	0.3	.5
(?)	4	.6	.4	.3	.8
β-Myrcene	5	.9	.8	.6	.8
Heptanal	6	2.2	1.2	1.9	1.6
D-Limonene	7	1.8	1.2	1.3	1.9
Octanal	8	14.6	21.6	27.8	37.6
Methyl heptenone	9	.6	.8	1.6	1.3
Nonanal	10	1.3	2.0	3.8	4.0
Methyl heptenol	11	.9	1.2	1.9	1.9
(?)	12	1.9	.4	1.3	1.3
Octyl acetate	13	2.8	.8	1.6	1.3
Citronellal	14	1.3	1.2	.6	.8
Decanal	15	8.4	15.5	20.6	22.7
Linaloöl	16	10.3	4.9	....	....
Octanol	17	2.2	3.2	3.1	2.4
Linalyl acetate	18	5.0	2.8	4.1	3.2
Nonyl acetate	19	1.6	.8	3.8	3.2
Undecanal	20	13.4	12.7	8.4	5.6
Nonanol	21	4.1	2.8	.9	.3
Geranyl acetate	22	1.6	1.2	.6	.3
Decyl acetate	23	1.3	1.6	.9	.3
Decanol	24	2.2	.8	.6	.3
Dodecanal	25	8.4	7.7	4.7	2.9
Citral	26	.9	1.2	.9	.8
Terpineol	27	4.0	4.0	3.1	1.6
Carvone	28	4.0	4.8	2.8	1.6
Neryl acetate	29	.9	1.2	.9	.5
Nerol	30	tr	tr	tr	tr
Geraniol	31	tr	tr	tr	tr

*From Kesterson and Hendrickson (1963).
†Compounds are stated as composition percentages. Figures represent peaks.

order to determine the oxygenated constituents in these oils, the terpeneless fractions were prepared by the method of Kirchner and Miller (1952). As a result of these analyses, the data showed that the concentrations of octyl aldehyde, nonyl aldehyde, methyl heptenol, and decyl aldehyde increased as the fruit matured, and the concentrations of linaloöl, undecyl aldehyde, nonyl alcohol, geranyl acetate, decyl alcohol, dodecyl aldehyde, and terpineol decreased with fruit growth and development. The concentrations of octyl, nonyl, and decyl aldehydes not only increased with the advance of the season, but they also accounted for 65 per cent of the oxygenated constituents in the terpeneless fractions prepared from the oils of the mature fruit. During the sampling period, the linaloöl content of the oil decreased from 10.3 per cent to undetermined amounts in the mature fruit. It was emphasized that the absence of linaloöl in mature Marsh grapefruit oil was the most important difference between grapefruit and orange oils.

To ascertain if varietal differences existed, Kesterson *et al.* (1965) determined the physical and chemical characteristics of Duncan grapefruit oils on samples collected when the fruit first reached legal maturity, and subsequently at 6-week intervals throughout the 1963–64 processing season, in order to follow the progressive effects of fruit maturity on oil quality. In table 114, the characteristics for each of the 6 oil samples can be compared and seasonal trends can be observed. In contrast to the characteristics of Marsh grapefruit oils (see table 112), the specific gravities, refractive indices, optical rotations, evaporation residues, acid numbers, percentages of free acid, ester values before acetylation, and free alcohol remained more or less constant throughout the processing season. The decreases in the amounts of total alcohol and ester after acetylation were attributed to aging of the oil samples rather than fruit maturity. As in the Marsh oils, the aldehyde content increased with fruit maturity, reaching a plateau in samples from February through May with a decreasing amount in the June sample. This result was contrary to what the same authors had recorded for cold-pressed Valencia orange oils (Kesterson and Hendrickson, 1953). They noted that aldehyde content was definitely associated with the flavor factor of the oil. Since there is a seasonal increase in both nootkatone and aldehyde contents with fruit maturity, it follows that the most appropriate time to recover grapefruit oil of optimum flavor is late in the processing season. (See under the subheading "Nootkatone," closely following.)

Varietal differences in the essential oils of grapefruit were also demonstrated by comparing the composition of terpeneless red and white grapefruit oils, as well as the physical and chemical properties of cold-pressed oils from both the red and white varieties (Kesterson and Hendrickson, 1964). The experimental materials used in these investigations were obtained from 3 red and 3 white grapefruit oils, selected from 3 different commercial plants

TABLE 114

PHYSICAL AND CHEMICAL PROPERTIES OF EXPRESSED DUNCAN GRAPEFRUIT OIL AS RELATED TO FRUIT MATURITY.*

Constituents and related data	Sample numbers and processing dates					
	1 11/19/63	2 12/31/63	3 2/11/64	4 3/24/64	5 5/5/64	6 6/16/64
Specific gravity (25°/25° C.)	0.8538	0.8518	0.8517	0.8514	0.8515	0.8531
Refractive index n_D^{20}	1.4765	1.4761	1.4760	1.4759	1.4761	1.4764
Refractive index 10 per cent distillate n_D^{20}	1.4714	1.4716	1.4717	1.4718	1.4720	....
Difference	0.0051	0.0045	0.0063	0.0041	0.0041	....
Optical rotation $[\alpha]_D^{25}$	+92.77	+92.97	+93.17	+93.57	+93.13	+92.71
Optical rotation 10 per cent distillate $[\alpha]_D^{25}$	+97.64	+97.84	+98.24	+98.64	+98.55	....
Difference	+4.87	+4.87	+5.07	+5.07	+5.42	....
Aldehyde content (per cent)	1.43	1.62	1.80	1.76	1.79	1.61
Evaporation residue (per cent)	7.89	6.87	7.48	7.25	7.26	8.42
Acid number	1.33	1.14	1.54	1.46	1.40	1.66
Free acid (per cent)	0.34	0.29	0.39	0.38	0.36	0.43
Ester number before acetylation	10.88	9.97	9.34	8.51	9.36	9.62
Ester content before acetylation (per cent)	3.80	3.48	3.26	2.97	3.27	3.36
Ester number after acetylation	15.45	13.33	13.17	13.24	13.08	12.65
Ester content after acetylation (per cent)	5.40	4.66	4.60	4.63	4.57	4.42
Free alcohol (per cent)	1.26	0.93	1.06	1.30	1.03	0.83
Total alcohol (per cent)	4.24	3.66	3.62	3.64	3.59	3.48

*From Kesterson *et al.* (1965).

that used either screw presses or FMC In-Line Extractors for oil recovery. Each sample consisted of 1 red and 1 white grapefruit oil, which represented the total seasonal production of each type of oil from each plant. Table 115 shows that the values for specific gravity, refractive index, difference between original oil and 10 per cent distillate for refractive index and optical rotation, and evaporation residue were higher without exception for the red grapefruit oils, while the values for optical rotation and aldehyde content were lower than those for white grapefruit oils.

It should be borne in mind that optical rotation is a specific property of citrus oils from all species.

The approximate concentrations of various constituents of the terpeneless oils of red and white grapefruit are reported in table 116. Kesterson and Hendrickson (1964) applied gas chromatographic methods to the terpeneless oil because this fraction was better adapted to these procedures for determining the oxygenated constituents in the absence of the naturally high percentage of terpene in cold-pressed oil. Their data showed that the chemical composition of the oils was highly variable, and that the 2 chief components present were octyl and decyl aldehydes. In white grapefruit oils, the ratios of octyl to decyl aldehydes were found to range from 1:1.1 to 1:1.4. In red grapefruit oils, the ratios were reversed, and ranged from 1.2:1 to 1.3:1. The red grapefruit oils contained a significant amount of linaloöl, while this constituent was absent from white grapefruit oils. It has been suggested that the differentiation between red and white grapefruit oils can be ascertained by the ratio of octyl to decyl aldehyde and by the presence (red) or absence (white) of linaloöl.

Terpenes. Terpenes are hydrocarbons of the formula $(C_5H_8)_n$, and occur widely distributed in nature. They are the chief constituents of the essential oils obtained by distilling with steam the sap and tissues of certain plants, particularly those of the coniferous and citrus groups. Essential or ethereal oils prepared in this way are used in the production of perfumes and in pharmacy. There occur with the terpenes closely related compounds containing oxygen, and these have been considered to be substitution products of the terpenes, such as alcohols and ketones. Hydrocarbons of this group are generally subdivided into hemiterpenes, C_5H_8 ("isoprene"); terpenes, $C_{10}H_{16}$; sesquiterpenes, $C_{15}H_{24}$; and diterpenes. The terpenes, $C_{10}H_{16}$, are all unsaturated hydrocarbons containing either 1 or 2 ethylene bonds in the molecule. In this class of compounds are limonene and terpinene. Dicyclic terpenes contain 1 double bond in the molecule. The chemical structure consists of 2 rings, a combination of a hexamethylene with a tetramethylene ring. Compounds of this type are pinene and camphene.

The essential oils of citrus fruits are composed chiefly of hydrocarbons designated as terpenes ($C_{10}H_{16}$) and of considerably smaller amounts of ses-

TABLE 115

PHYSICAL AND CHEMICAL PROPERTIES OF COLD-PRESSED WHITE AND RED GRAPEFRUIT OILS.*

Constituents and related data	FMC In-Line Extractor				Screw press	
	White		Red		White	Red
Specific gravity (25°/25° C.)	0.8539	0.8539	0.8604	0.8576	0.8534	0.8552
Refractive index n_D^{20}	1.4759	1.4761	1.4786	1.4778	1.4759	1.4766
Refractive index 10 per cent distillate n_D^{20}	1.4719	1.4719	1.4718	1.4719	1.4718	1.4718
Difference	0.0040	0.0042	0.0068	0.0059	0.0041	0.0048
Optical rotation $[\alpha]_D^{25}$	+92.87	+93.27	+90.67	+90.87	+92.67	+91.07
Optical rotation 10 per cent distillate $[\alpha]_D^{25}$	+97.02	+97.22	+96.82	+97.02	+97.04	+96.84
Difference	+4.15	+3.95	+6.15	+6.15	+4.37	+5.77
Evaporation residue (per cent)	6.69	5.99	9.39	8.02	6.32	7.12
Aldehyde content (per cent)	1.69	1.42	1.23	1.23	1.56	1.38

*From Kesterson and Hendrickson (1964).

quiterpenes ($C_{15}H_{24}$). These 2 components, together with the oxygenated compounds, such as alcohols, aldehydes, ketones, acids, and esters, are the constituents of oils that contribute the characteristic odor to citrus products.

Stanley *et al.* (1961*a*) described a method of separating the terpene hydrocarbons from citrus oils, with subsequent analysis by gas chromatography. Although their data for the orange are not reported here, the chromatogram

TABLE 116

THE COMPOSITION OF TERPENELESS WHITE AND RED GRAPEFRUIT OILS.*

Compounds†	FMC In-Line Extractor				Screw press	
	White		Red		White	Red
α-Pinene	0.75	0.90	0.29	0.45	0.47	0.62
(?)	.37	.72	.36	.23	.05	.04
(?)	.37	.72	.36	.36	.09	.42
β-Myrcene	.75	.90	.71	1.13	.14	1.66
Heptanal	2.62	1.44	.89	1.35	.23	1.04
D-limonene	3.75	2.71	2.14	1.35	.70	3.12
Octanal	17.99	19.51	20.52	25.66	29.52	25.81
(?)	.75	2.35	1.07	.68	.94	1.04
Methyl heptenone	2.25	2.35	2.14	.90	.94	1.46
Nonanal	4.49	5.23	4.10	3.15	3.75	3.75
Methyl heptenol	1.87	1.44	1.78	.90	1.17	.83
(?)	.37	.90	.54	.45	.70	.62
Octyl acetate	4.12	4.15	4.10	2.48	1.64	2.70
Citronellal	1.50	1.08	1.43	1.13	.42	1.25
Decanal	25.85	26.01	16.41	21.38	31.87	20.81
Linaloöl	....	....	3.92	2.93	....	2.08
Octanol	1.12	1.62	1.25	.27	.70	1.25
Linalyl acetate	2.25	3.61	2.50	2.25	2.11	2.91
Nonyl acetate	3.37	4.87	4.46	1.58	3.05	2.08
Undecanal	5.99	4.69	4.82	3.38	4.92	4.16
Nonanol	.75	.36	.36	.45	.37	.42
Geranyl acetate	1.87	.90	1.78	.41	.47	.83
Decyl acetate	1.12	.72	.71	.09	.14	.29
Decanol	1.87	1.81	1.78	1.13	.19	.42
Dodecanal	3.00	3.61	4.46	6.76	3.28	4.37
Citral	2.62	3.25	3.57	4.28	2.11	1.66
Terpineol	3.00	3.07	4.10	7.21	6.33	6.66
Carvone	4.49	.90	7.31	5.86	2.34	5.41
Neryl acetate	.75	.18	2.14	1.80	.14	1.25
Nerol	tr	tr	tr	tr	.05	.83
Geraniol	tr	tr	tr	tr	1.17	.21
Ratios: Octyl:decyl	1:1.4	1:1.3	1.3:1	1.2:1	1:1.1	1.2:1

*From Kesterson and Hendrickson (1964).
†Compounds are stated as composition percentages.

for grapefruit terpenes (figure 68) resembles the pattern distribution of orange-oil terpenes. More than 95 per cent of the terpene fraction of grapefruit oil is D-limonene.

The discussion that follows is concerned with certain factors that affect the optical rotation of lemon oils. These data, although they were experimentally obtained on lemon oils, help to clarify the reason for the high optical rotation of grapefruit oils.

Stanley and his fellow experimenters determined that large variations in the optical rotation of lemon oils occur, and that these variations in California lemons are related to the districts in which the fruit is grown. The optical rotation of lemon oils from inland districts may be as high as 70°, whereas samples from coastal districts may fall below 50°. Attention was drawn to the frequent suggestion that changes in optical rotation are caused by L-limonene as well as D-limonene. When their values for optical rotation are examined in relation to the terpene data, it can be seen that the large variations in optical rotation were highly correlated with variations in the relative proportions of β-pinene to D-limonene.

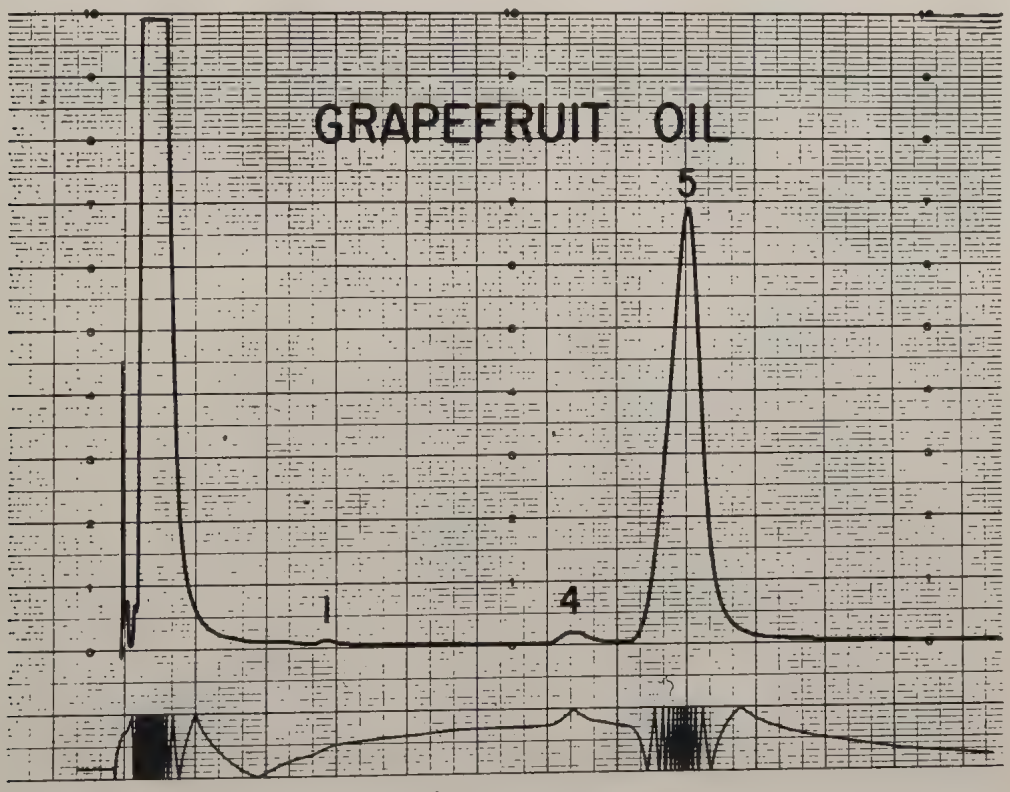


Fig. 68. Chromatograph of grapefruit-oil terpenes. Column conditions: 80-microliter sample; 85° C. (185° F.); 120 milliliters helium per minute; thermal conductivity detector, 10-foot × one-fourth-inch column (3.05 meters × 7 mm); 20 per cent diethylene glycol succinate on 60 to 80-mesh firebrick. Length of time for complete chromatograph, about 15 minutes. (From Stanley *et al.*, 1961*a*.)

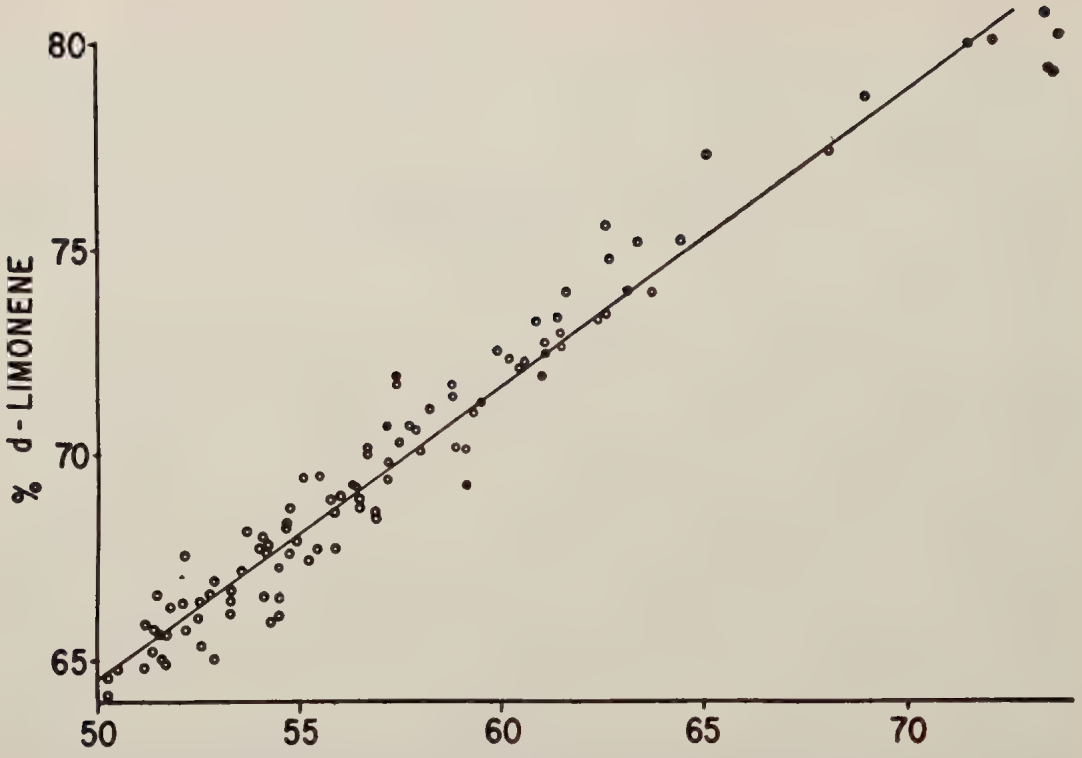


Fig. 69. Plot of D-limonene percentage against optical rotation. Open circles indicate domestic samples; black circles, foreign samples. (From Stanley *et al.*, 1961*a*.)

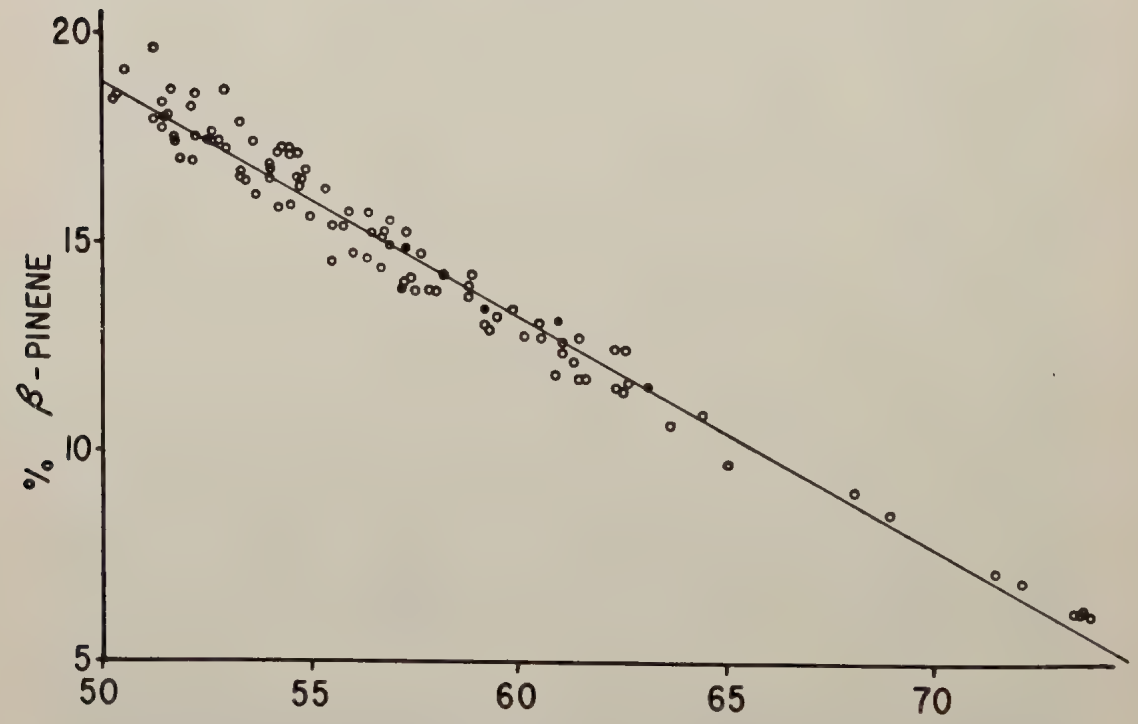


Fig. 70. Plot of *beta*-pinene percentage against optical rotation. Open circles indicate domestic samples; black circles, foreign samples. (From Stanley *et al.*, 1961*a*.)

These relationships are clearly demonstrated in figures 69 and 70. Based on the degree of scattering of the data, it will be observed that a high positive correlation resulted when the concentration of D-limonene was plotted against the optical rotations. In contrast to lemon oil, grapefruit oils have optical rotation values above 90°, and since more than 95 per cent of the terpene fraction is D-limonene, it would be reasonable to assume that the optical rotation of grapefruit oil is highly correlated with D-limonene.

The plot of β-pinene content against optical rotation resulted in a highly negative correlation. It was found that the relationship between terpene composition and optical rotation was applicable to both foreign and domestic lemon oils.

Fisher and Nordby (1965), in studying the composition of citrus products, isolated 9 coumarins from Florida grapefruit-peel oil, and made spectral characterizations of them (table 117). Five of these compounds—bergamottin, limettin, bergapten, and 2 formylated coumarins—had not been reported earlier in grapefruit. Other investigators (Stanley and Vannier, 1957*a*, *b*; Peyron, 1963; Stanley, 1963; and Stanley *et al.*, 1965) reported the separation and characterization of many coumarins in citrus.

Weizmann *et al.* (1955) found β-sitosterol in grapefruit-peel oil, and Weizmann and Mazur (1958) found a new sterol, citrostadienol, in grapefruit oils.

Nootkatone. In studying the flavor constituents of grapefruit oils, MacLeod and Buigues (1964) reported the presence of a bicyclic conjugated sesquiterpene ketone, nootkatone, in grapefruit-peel oil and in grapefruit juice that was known to be free of peel oil. Nootkatone has a carbon structural frame-

TABLE 117

COUMARINS AND PSORALENS SEPARATED ON SILICA GEL AND OBTAINED FROM THE VARIOUS HEXANE–ETHYL ACETATE ELUATE FRACTIONS.*

Compounds isolated	Compound numbers	Fractions combined	Total fraction volume (liters)	Ethyl acetate in eluate (per cent)
Bergamottin	I	18–24	6.0	2.5
7-Geranyloxycoumarin	VI	25–59	40.0	2.5
Osthol	IV	60–70	12.0	2.5
Limettin	V	89–99	8.0	5.0
Bergapten	III	100–110	12.0	5.0
5-[(3,6-dimethyl-6-formyl-2-heptenyl)oxy]-psoralen	...	128–136	9.0	7.5
7-methoxy-8-(2-formyl-2-methylpropyl)-coumarin	...	159–167	10.0	10.0
Bergaptol	II	189–190	2.5	100.0
7-[(6,7-dihydroxy-3,7-dimethyl-2-octenyl)oxy]-coumarin	...	191	1.0	100.0

*From Fisher and Nordby (1965).

work identical to valencene, an orange-oil hydrocarbon, but they differ in composition in that valencene is lacking 1 oxygen atom. A constituent which corresponded to an unidentified GLC (gas-liquid chromatographic) peak in cold-pressed grapefruit oil was isolated and identified as nootkatone (figure 71).

The identification was determined by its melting point, infrared spectrum, and the melting point of its 2,4-dinitrophenylhydrazone derivative.

This ketone is a colorless, low-melting, crystalline solid which has an astringent taste and a musty odor. In grapefruit juice, nootkatone occurs in concentrations equivalent to approximately the threshold of taste detection, while in grapefruit-peel oil, it ranges as high as 3,000 ppm (0.3 per cent). The results of taste-panel studies indicate that the threshold taste detection values of nootkatone in an aqueous sucrose solution (32° Baumé) range between 20 and 40 ppm. The odor of nootkatone is detectable below 10 ppm. MacLeod and Buigues drew attention to the fact that Kirchner and Miller (1953) had reported the first indication of an unknown sesquiterpene ketone in grapefruit juice, but concluded from their own researches on the oily extract from grapefruit juice that the sesquiterpene ketone that Kirchner and Miller isolated was probably nootkatone.

MacLeod (1966) reported on his survey of the nootkatone content of grapefruit oils obtained from various places throughout the world. The content in these samples varied from less than 0.01 per cent to 1.8 per cent for late-season California oils. In the oil samples, the increase in flavor intensity was associated with an increase in the nootkatone content.

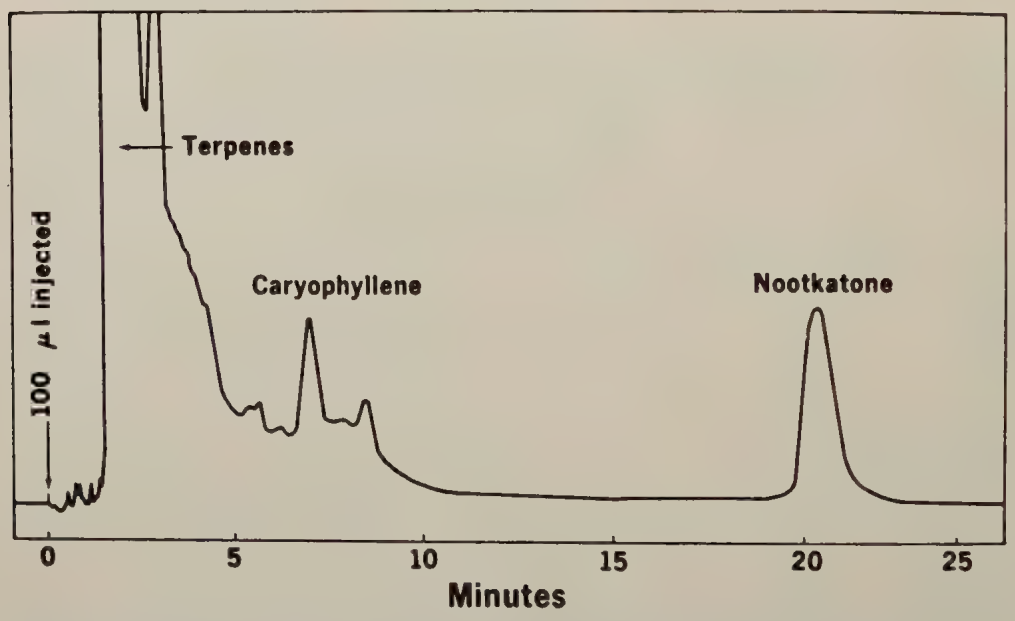


Fig. 71. Gas chromatogram of high-quality grapefruit oil on the one-quarter-inch (7-mm) column at 215° C. (419° F.) (From MacLeod and Buigues, 1964.)

If the nootkatone content of grapefruit-peel oil is related to good flavor, it is essential to determine to what extent the concentration of this constituent varies with fruit maturity. Kesterson *et al.* (1965) collected Duncan grapefruit samples (on rough-lemon rootstock) when the fruit first reached legal maturity, and subsequently at 6-week intervals throughout the 1963–64 processing season, for the specific purpose of determining the nootkatone content of the expressed grapefruit oil. The nootkatone content increased from 0.065 to 0.810 per cent during the growth and maturity of the fruit, while the oil yield decreased from 2.55 to 0.50 pounds per ton of fruit under these same experimental conditions (table 118). Reference has already been made to the fact that the aldehyde contents of these oil samples increased significantly with the advance of the season. Because the nootkatone content showed more than a 10-fold increase during the sampling period, the inference was drawn that this constituent could probably be used as an index of fruit maturity. Furthermore, the data substantiated the fact that nootkatone and aldehyde contents were highly associated with good flavor, indicating that the cold-pressed oil should be recovered late in the processing season.

Hunter and Brogden (1965*a*) separated and identified the terpene and sesquiterpene hydrocarbons in the essential oils of the juice and peel of Duncan grapefruit. These hydrocarbons were analyzed in 2 steps. The first step consisted in removing the oxygen-containing compounds by column chromatography, and the second step consisted in analyzing the terpenoid-free oil by gas chromatography, using a small sample for terpene analysis and a larger sample for sesquiterpene analysis. The constituents were identified and determined by infrared and mass spectroscopic techniques. The constituents represented by each peak are identified on the graphs of figures 72 and 73. Duncan peel and juice oils showed variations in the amounts of constituents in that there was only 25 per cent as much ylangene in the juice

TABLE 118

OIL YIELD AND NOOTKATONE CONTENT OF EXPRESSED DUNCAN GRAPEFRUIT OIL.*
(These oil samples were prepared at the University of Florida Citrus Experiment Station pilot plant, utilizing the FMC In-Line Extractor with mist-spray attachment.)

Processing dates	Sample numbers	Fruit weight (pounds)†	Oil quantity (milliliters)	Oil yield (pounds per ton)†	Nootkatone (per cent)
11/19/63	1	1,510	1,025	2.55	0.065
12/31/63	2	1,530	975	2.38	.285
2/11/64	3	1,386	465	1.25	.503
3/24/64	4	1,405	500	1.33	.693
5/5/64	5	1,286	425	1.23	.750
6/16/64	6	1,017	135	0.50	.810

*Adapted from Kesterson *et al.* (1965).
†One pound equals 0.454 kilogram. One ton (American) equals 2,000 pounds.

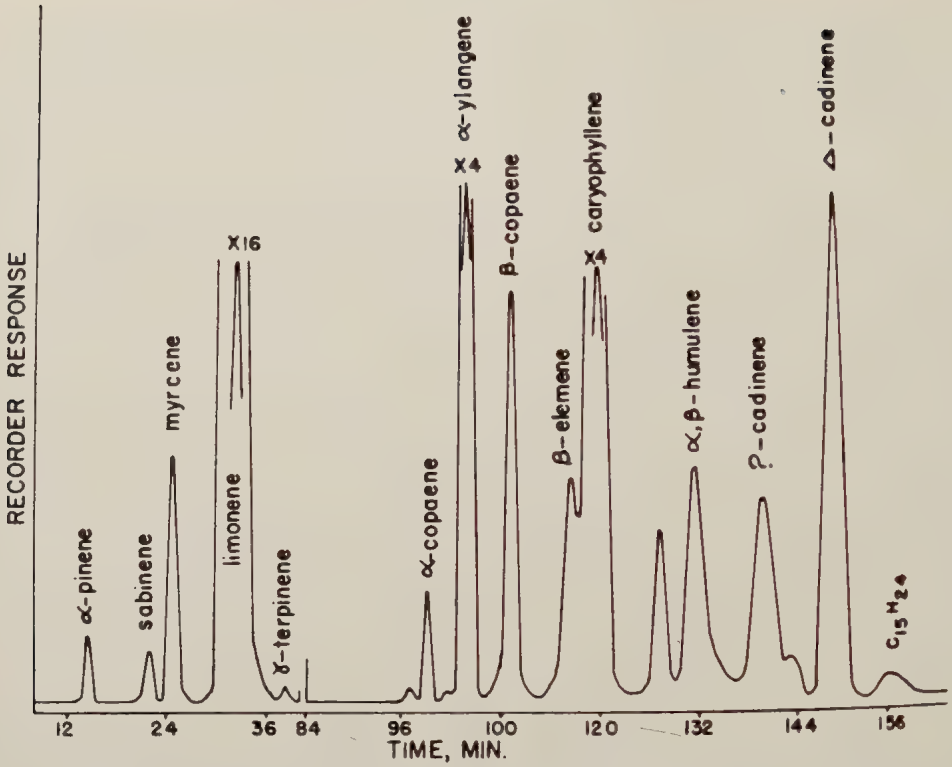


Fig. 72. Analyses of the terpene and sesquiterpene hydrocarbons of the essential oils of Duncan grapefruit peel. (From Hunter and Brogden, 1965*a*.)

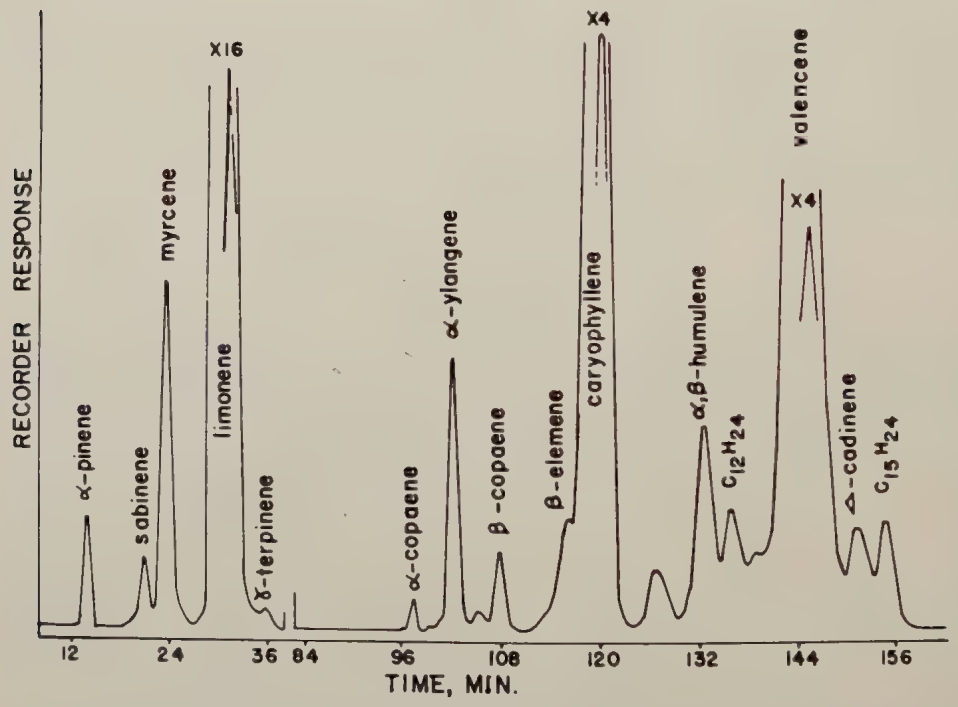


Fig. 73. Analyses of the terpene and sesquiterpene hydrocarbons of the essential oils of Duncan grapefruit juice. (From Hunter and Brogden, 1965*a*.)

oil as in the peel oil. Valencene, the principal sesquiterpene constituent of the juice oil, was not reported in the peel oil. The juice oil contained a considerably lower concentration of β-copaene than the peel oil.

The close similarity in the chemical structures of valencene and nootkatone was observed by Hunter and Brogden (1965*b*), who converted valencene into nootkatone with tertiary butyl chromate, an oxidizing reagent which reacts selectively at the allylic position. This experiment was performed on valencene that had been isolated from orange oil and subsequently oxidized, with the reagent, to the sesquiterpene ketone having the same physical properties as the natural nootkatone. There synthetic nootkatone was identical to the product isolated from grapefruit essential oils.

Aldehydes. Those who have analyzed the essential oils of citrus have found that the aldehydes are present in only very small quantities, especially in grapefruit oil. Nevertheless, they are of importance, because they possess characteristic odors and flavors.

Nelson and Mottern (1934), in their analysis of grapefruit oil mentioned earlier, found from 1.60 to 1.74 per cent octyl and decyl aldehydes with an average of 1.67 per cent (table 111). From 16 liters of oil they obtained a total of 117 grams of aldehydes. The following other substances were identified: geraniol and octyl alcohol (both free and as acetates), cadinene, and small quantities of citral and methyl anthranilate.

Biale and Weiss (1939) distilled 615 grams of ground fresh grapefruit peel 4 times, and found the following amounts of aldehydes.

Fraction	*m.e.*
1	0.391
2	0.053
3	0.030
4	0.030
Total	0.504

(equals 0.084 m.e. per 100 grams of peel)

The rapid drop in amount of aldehydes with successive distillations suggested to these workers that most of the aldehyde was produced in the peel during respiration, and not by the breakdown during distillation.

Biale and Shepherd (1939) stated that acetaldehyde could be found in citrus fruits (grapefruit, lemons, and oranges) under storage conditions where ventilation was restricted. Of the aldehydes given off by the fruit, acetaldehyde predominated.

Kesterson and McDuff (1948) and Hendrickson and Kesterson (1965) reported the following amounts of aldehydes in grapefruit-peel oil: cold-pressed, 1.49 to 1.67 per cent; distilled, 2.30 to 4.06 per cent. Steam-distilled oil obtained in the deoiling of grapefruit juice was 90 per cent higher in aldehydes and 60 per cent lower in esters than the press oils.

TABLE 119

QUANTITATIVE ANALYSES OF THE MAJOR ALDEHYDES IN COLD-PRESSED ORANGE, GRAPEFRUIT, AND LEMON OILS.*

Citrus oils	Aldehydes (per cent)							
	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	Carvone†	Citral
Valencia orange (California).....	2.6	38.6	5.4	41.8	1.8	7.9	2.0	....
Navel orange (California).....	3.8	26.9	5.5	40.2	2.9	12.1	8.6	....
Temple orange (Florida).....	1.5	48.6	2.2	34.1	9.3	4.2	...	....
Grapefruit, white (Arizona).....	3.4	34.7	6.4	43.4	2.5	7.7	...	....
Grapefruit, pink (Arizona).....	3.9	16.4	8.8	53.9	6.0	10.9	...	....
Lemon (California)‡.....	1.1	3.7	5.6	2.8	2.1	...	...	84.6

*From Stanley *et al.* (1961*b*).
†Retention time was identical with that of a sample of carvone.
‡Total carbonyl, 2.6 per cent, by the method of Fritz *et al.* (1959).

Stanley *et al.* (1961*b*) developed and perfected analytical procedures for determining the relative concentrations of the normal saturated aldehydes and citral from lemon, orange, and grapefruit cold-pressed oils. Emphasis is placed here on the grapefruit data.

The results reported by Stanley and co-workers in table 119 compare the major aldehydes of grapefruit oils with those of the lemon and orange. These are typical, analytical data, determined on cold-pressed oils from citrus fruits, and the values were obtained with procedures specifically adapted to lemon oils The highest concentrations of aldehydes in these oils consisted principally of C_8 (N-octanal) and C_{10} (N-decanal) constituents. In the grapefruit data, C_7, C_9, C_{11}, and C_{12} aldehydes existed in concentrations as high as 10.9 per cent of the total aldehydes. The gas chromatogram of major aldehydes of grapefruit oils isolated by the method of Girard and Sandulesco (1936) is shown in figure 74. Note that this tracing was not used for quantitative determinations. The normal saturated aldehydes were the only ones reported for cold-pressed grapefruit oils. It was also noted that other carbonyls were present which apparently were not completely regenerated under the conditions established for lemon oil. (Numerals in figure represent minutes.)

Chemical Stability of Peel Oils. A voluminous literature has accumulated on the factors affecting the stability of peel oils, but no attempt will be made here to make a historical evaluation of the published data on all the variables

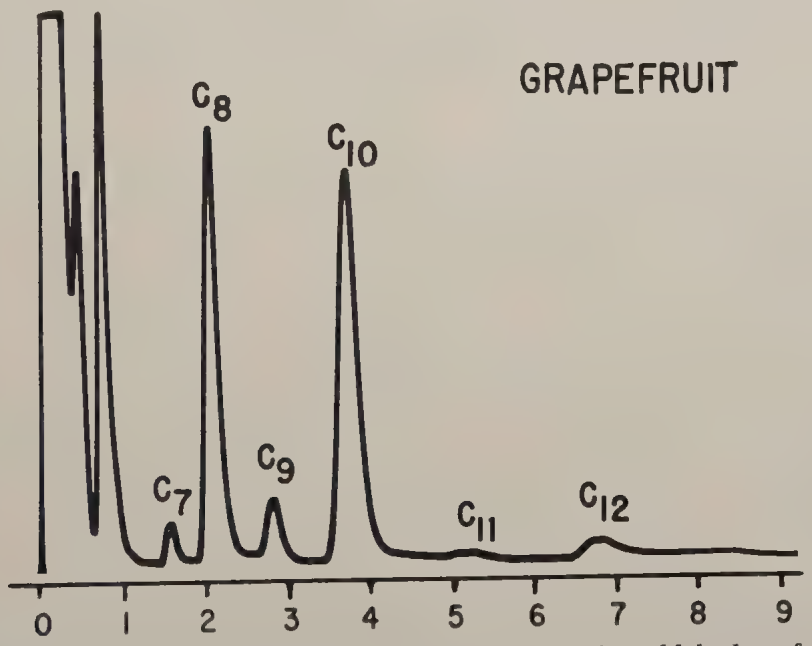


Fig. 74. Gas chromatographic analysis of major aldehydes of grapefruit oils, isolated by the method of Girard and Sandulesco (1936). Column conditions: Temperature, 200° C. (392° F.); 20 milliliters helium per minute; 10-foot × one-fourth-inch column (3.05 meters × 7 mm); butane diol succinate stationary phase on crushed firebrick. (From Stanley *et al.*, 1961*b*.)

affecting the quality of citrus-peel oils. Our purpose is to consider the factors that affect the chemical stability of these oils, and to relate the data to the characteristics of the essential oils of grapefruit peel.

It has been well established that the oil content of citrus fruits increases with fruit maturity (Hood, 1916), and that the effects of age, size, variety, and environmental factors are highly correlated with the oil content of the peel (Bartholomew and Sinclair, 1946). Apparently, the increases in the oil contents of citrus fruit peels follow a pattern similar to the changes that occur in the soluble sugars and total soluble solids of the fruit during growth and maturation. With all the metabolic changes that occur in the fruit during the growth cycle, some changes in the chemical composition of the oils should also take place, concurrently with the changes that produce an increase in the total oil content of the peel.

Hendrickson and Kesterson (1965) noted that the yield of oil obtained from the peel had the greatest effect on the physical and chemical properties of cold-pressed orange oils. Higher oil yields were associated with increased values in specific gravity, evaporation residues, and refractive indices, but they were negatively correlated with decreased values in optical rotation (figures 75 and 76). As would be expected, the higher oil yields contained higher molecular-weight constituents with correspondingly higher boiling points. (See also Kesterson and Hendrickson, 1953.)

The lower optical rotation values were undoubtedly caused by the dilution of D-limonene with the addition of constituents to the oil component as a result of higher oil yields.

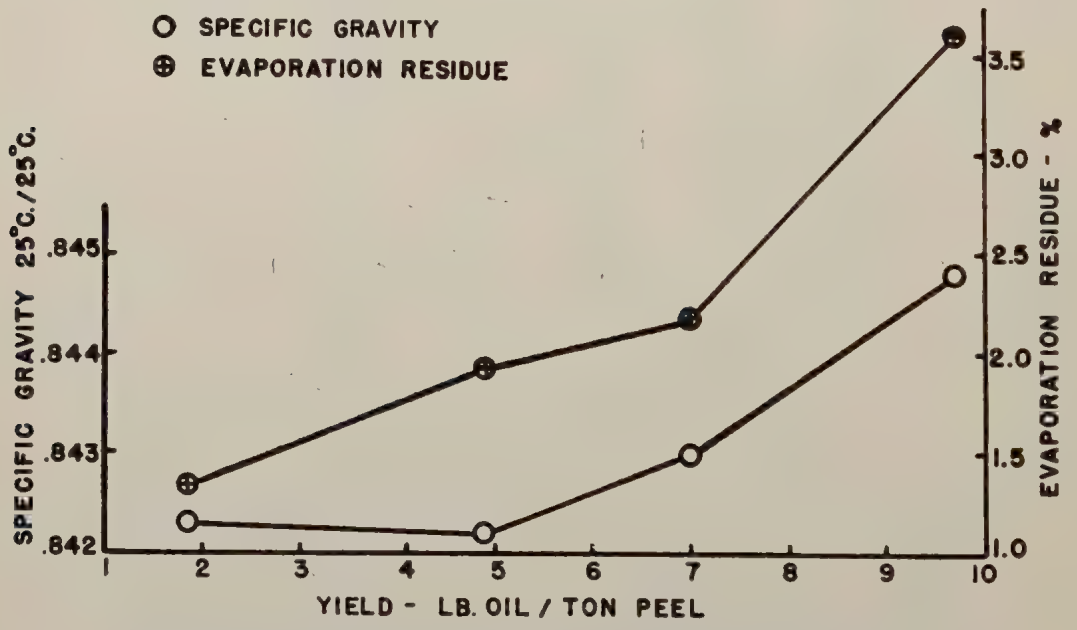


Fig. 75. Relationships of specific gravity and evaporation residue of cold-pressed orange oils to yield. (From Kesterson and Hendrickson, 1953.)

These investigators observed the relation between the aqueous phase used in oil production and the aldehyde content of the oil. It was noted that under constant processing conditions, the reduction in use of water from large quantities to amounts equivalent to 100 gallons (379 liters) per gallon of oil produced an increase in the aldehyde content from 1.08 to 1.64 per cent, about 52 per cent.

The essential oils of citrus fruits are subject to oxidation in the presence of atmospheric oxygen. The oxidative reactions are accelerated by heat, moisture, and certain metals, the presence of which catalyzes the reactions. The sources of the off-flavors and odors of orange oils have been attributed to the oxidative changes that occur in the samples during storage. Consequently, the origin of the "terpeney" flavor in the essential oils of citrus peels has also been attributed to oxidative reactions. Control of these reactions can be effected by the use of legally permitted antioxidants, and by replacement of the air in the oil container with carbon dioxide.

Kesterson and McDuff (1949) and Proctor and Kenyon (1949) attempted to correlate the stability of orange oils with the relative rates of oxygen absorption. If these estimates were correct, it follows that the longer the time required for absorption of a given quantity of oxygen, the more stable is the oil toward oxidative spoilage.

After demonstrating that citrus-peel oils evolved gas by the manometric procedure used for oxygen absorption, Kesterson and Hendrickson (1951) developed an improved manometric method by using Ascarite to absorb the evolved carbon dioxide and water from the system, thus permitting the de-

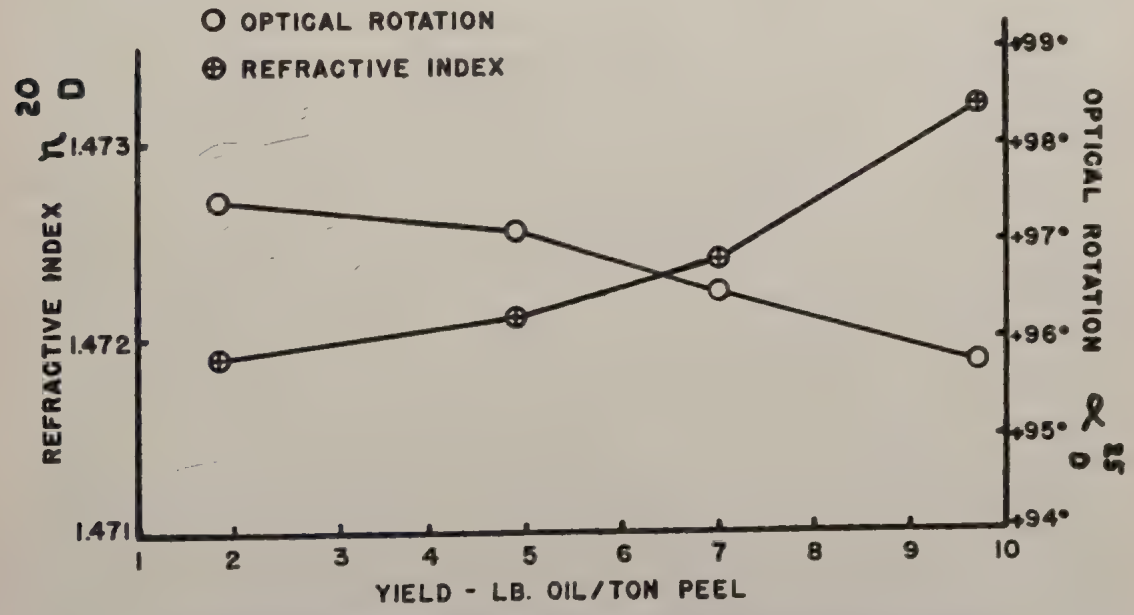


Fig. 76. Relationships of refractive index and optical rotation of cold-pressed orange oils to yield. (From Kesterson and Hendrickson, 1953.)

termination of the true oxygen uptake by cold-pressed orange oil. Their data showed the relative stabilities of the various oils in terms of time required for a 1-gram sample to absorb 1,200 microliters (1.2 milliliter) of oxygen (figure 77). At this level of oxygen uptake, the oils were "terpeney" in aroma and lighter in color.

Since the physical and chemical properties of cold-pressed citrus oils are characteristically influenced by the oil yields of the peel, it should follow that oxygen absorption and methods of extraction will influence oil stability. This generalization is well illustrated in figure 77, which shows the relative oxygen absorption rates of cold-pressed Valencia orange oils exposed to an atmosphere of oxygen at 40° C. (104° F.) for various lengths of time. Note that the method of extraction markedly affected the rate of oxygen absorption of the oil. The 3 procedures, using the Pipkin Roll, Pipkin Juice, and Fraser-

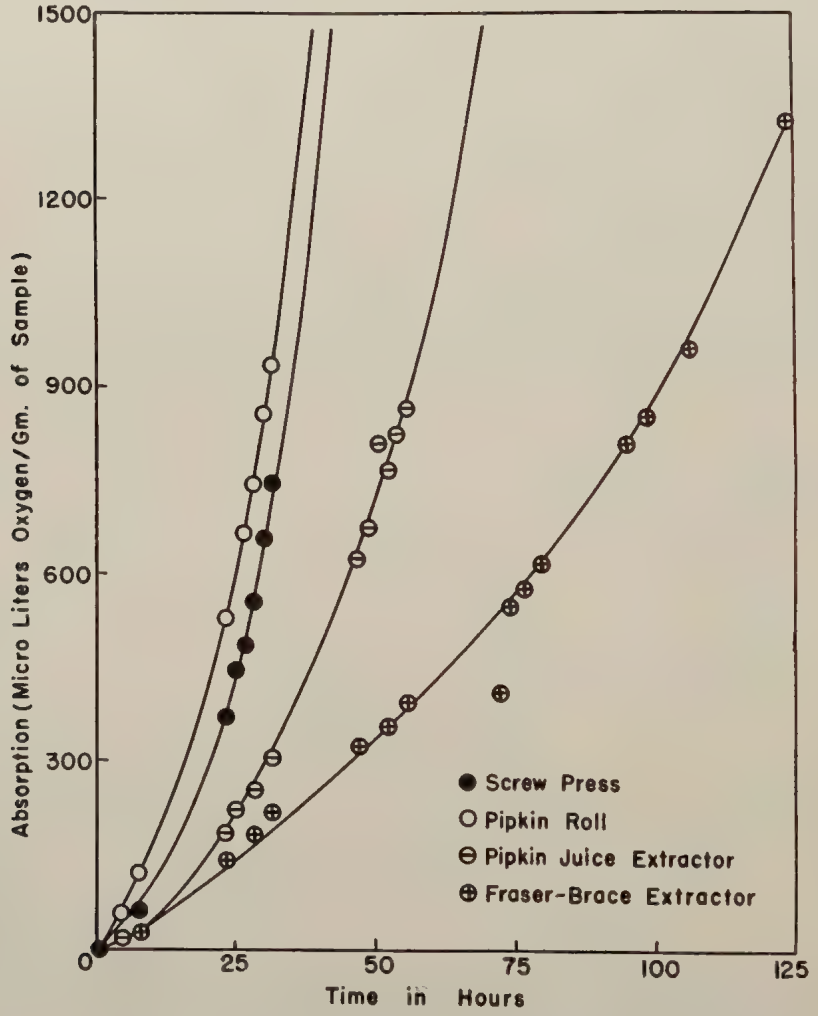


Fig. 77. Oxygen uptake (in microliters) of Florida cold-pressed Valencia orange oils when exposed to an atmosphere of oxygen at 40° C. (104° F.). (From Kesterson and Hendrickson, 1951.)

Brace extractors, are listed in the order of increasing oil yield, which characteristic shows a direct correlation with stability. The oil extracted by the screw-press method behaved differently; the oil yields were higher than those from the Pipkin Roll extractor. Furthermore, this oil had a higher rate of oxygen absorption than those from the Pipkin Juice and Fraser-Brace extractors, thus decreasing its oxidative stability. Since the screw-press oil was obtained by crushing the peel against a perforated screen, Kesterson and Hendrickson (1953) postulated that in the extraction process, the albedo might have absorbed a naturally occurring antioxidant from the oil, thus reducing its stability by increasing the relative rate of oxygen uptake.

Distilled Oils. One of the chief characteristics of citrus-fruit oils is their volatility with steam. This property is advantageous where distillation with hot water or steam is carried out *in vacuo* at temperatures lower than the boiling point of water—212° F. or 100° C. Distilled oils of oranges and grapefruit are obtained as by-products in the canning of juices. Mechanical extractors usually incorporate excessive amounts of peel oil in the juice, which must be reduced to a low concentration. To maintain maximum flavor and storage life, the juice should contain a designated amount of recoverable oil. To remove excessive amounts of oil, the juice is passed into a deoiler, where it is flashed under a vacuum of 11 to 26 inches (28 to 66 centimeters) and 190 to 130° F. (88 to 54° C.). The vaporized oils and water are condensed, and on standing, the condensate separates into the oil and water phases. Citrus oils that are steam-distilled under a vacuum have different properties from those that are steam-distilled at atmospheric pressures.

Stripper oils are obtained by steam distillation of citrus press liquor, which is subsequently used in the production of citrus molasses. It is possible to recover 60 to 80 per cent of the available oil in citrus press liquor by flashing the liquor from 240° F. (116° C.) to its atmospheric boiling point.

Hendrickson and Kesterson (1965) estimated Florida's potential production to be over 10 million pounds (4.54 million kilograms) of stripper oil per year. This has never been practically realized because many processors are not equipped to recover stripper oil, and molasses production is often avoided, thus resulting in an annual production of oil that is considerably below the potential. Stripper oil is composed of a mixture of steam-distilled oils obtained from the press liquor that has been derived from a mixture of citrus fruits.

The values reported in table 120 were taken from the data of the above authors on the physical and chemical properties of both distilled and stripper oils of Florida oranges and grapefruit. They noted that these values for distilled oils were typical for distilled oils of high quality. The orange oils averaged higher aldehyde contents (24 per cent) and lower ester values (10 per cent) than similar values for cold-pressed oils. In the deoiling process of grape-

TABLE 120

MAXIMUM AND MINIMUM VALUES FOR THE PHYSICAL AND CHEMICAL PROPERTIES OF DISTILLED AND STRIPPER CITRUS OILS.*

Constituents and related data	Orange† (11 samples)		Grapefruit† (9 samples)		Stripper oil (10 samples)	
	Maximum	Minimum	Maximum	Minimum	Maximum	Minimum
Specific gravity (25°/25° C.)	0.8464	0.8400	0.8539	0.8415	0.8433	0.8398
Refractive index n_D^{20}	1.4732	1.4715	1.4746	1.4714	1.4721	1.4713
Optical rotation $[\alpha]_D^{25}$	+98.56	+95.92	+96.50	+91.50	+98.90	+95.55
Aldehyde content (per cent)	2.48	1.15	4.06	2.30	1.50	0.47
Ester content (per cent)	1.38	0.22	2.52	0.08	2.46	.07
Evaporation residue (per cent)	1.24	.08	3.66	.19	0.79	.03

*Adapted from Hendrickson and Kesterson (1965).
†Aldehyde content: decyl.

fruit juice by flashing at high vacuum, the resulting steam-distilled oils averaged higher aldehydes (90 per cent) and lower ester values (60 per cent) than cold-pressed oils. Stripper oil is composed of more than 95 per cent D-limonene, and it serves as a good commercial source of this product.

Effects of Antioxidants on the Stability of Citrus Oils. An antioxidant is a chemical compound or a mixture of substances which, when added to fats, oils, and citrus-fruit oils (essential oils), chemically stabilizes the reaction against oxidative changes, thus preventing the formation of substances which are sources of off-flavors. Terpenes are unsaturated hydrocarbons, and are highly reactive with atmospheric oxygen. The terpene portion of citrus oils oxidizes into compounds which impart undesirable odors and flavors.

Considerable research has been published on the use of antioxidants to increase the stability of citrus-fruit oils. Lakritz (1943) found that hydroquinone had good antioxidant properties, and that a mixture of hydroquinone and wheat-germ oil was superior to either one in stabilizing lemon and orange oils. Kesterson and McDuff (1949) demonstrated the effectiveness of α-tocopherol as a stabilizing agent against oxidative changes in citrus oils. In studying the effects of antioxidants on orange oils, Kenyon and Proctor (1951) reported that the antioxidants that were effective in retarding oxidative changes in edible fats and oils were similarly effective in orange oils. They found α-tocopherol to be more effective than either nordihydroguaiaretic acid (NDGA) or propyl gallate. Guaiacol is highly effective as an antioxidant for orange oil, but it has a strong odor and is possibly toxic. Gearhart *et al.* (1957) determined the effectiveness of butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) in stabilizing D-limonene and orange oil against oxidative changes and deterioration. Both were effective, with the former being slightly better.

Data published by many scientists strongly indicate that the oxidative process of edible fats and oils is composed of 2 reaction periods: (1) the period of induction, and (2) the period of active oxygen absorption. During the period of induction, the oxygen absorption is at a minimum, and the susceptibility of a fat to oxidation is highly associated with the relative length of the period. There is great variation in the length of the induction periods among the various fats and oils, and there appears to be a marked correlation between the ease with which the various samples of fats develop odors and off-flavors and the shortness of the induction periods. The period of active oxygen absorption is that portion of the oxidative reaction which involves an extremely rapid rate of oxygen uptake by fatty acids, such as linoleic and linolenic, which are characteristic of the drying oils, and by those acids with a much slower rate of uptake, such as oleic and ricinoleic.

Off-flavor and/or rancidity are caused by 2 distinct chemical reactions which take place in fats and oils: (1) hydrolysis of the glycerides, with the

consequent formation of free fatty acids, and (2) oxidation of fats and oils which contain unsaturated acids, resulting in the formation of aldehydes, ketones, and acids of lower molecular weight than those originally present in the reaction medium. Oxygen is essential to carry out the oxidative type of rancidity. Both hydrolysis and oxidation may proceed simultaneously. From a practical standpoint, there is no sharp line of demarcation, but the 2 reaction mechanisms are chemically different.

To reason from the information in the foregoing paragraphs, it appears that certain properties in the oxidative process of edible fats and oils bear some similarities to the oxidative properties of essential oils of citrus fruits. For example, curves showing the rate of oxygen absorption by natural fats are not unlike the oxygen absorption curves of citrus-peel oils. Furthermore, both natural fats and essential oils of citrus peel show differences in the lengths of the induction periods of the various samples, as influenced by temperature, storage, and naturally occurring, as well as added, antioxidants. The shapes of the curves for oxygen absorption for both types of materials strongly indicate an autocatalytic process.

Newhall and Kesterson (1961) studied some of the factors that affected the autoxidation of D-limonene during storage. Their data confirmed the established principle that almost no change occurs in the physical properties of samples stored in the absence of air. On the contrary, samples that were continually exposed to the atmosphere gradually increased in refractive index, peroxide value, and acid number, and decreased significantly in optical rotation.

The recommendation of the authors for storage of D-limonene in drums consisted in filling the drums completely with hot D-limonene, adding 50 to 100 ppm of butylated hydroxytoluene (BHT), and excluding the air with nitrogen or carbon dioxide.

Autoxidation is inhibited in biological systems by natural antioxidants which occur as chemical constituents in the cellular material. Natural inhibitors that delay the reaction between fatty substances and atmospheric oxygen occur in fats and oils of biological origin in extremely small concentrations (approximately 0.1 per cent or less of the total fat and oil content). Reference has been made to various experiments of Kesterson and Hendrickson (1953). They found that high-yield extracting methods produced oils with relatively high stabilities by decreasing their rates of oxygen uptake, thus increasing the induction periods.

Charley (1963) reported that the increased flavor stability of comminuted base of whole fruit was attributed to pronounced antioxidant properties of some peel substance.

In studying the occurrence of natural antioxidants in citrus fruit, Ting and Newhall (1965) experimentally determined the relative amounts of anti-

oxidant substances in a number of citrus varieties, and estimated the antioxidant activity in various portions of them. Several extraction methods were used to isolate the active principle from the tissues, and a specially designed apparatus was used to measure the oxygen uptake of the samples. Since terpenes are the mostly easily oxidized substances in citrus oils, it is not surprising that D-limonene was used as an assay material for determining the comparative antioxidant activity of materials extracted from the tissues.

The data illustrated in figure 78 show the differences in the effects of extracts of component parts of Valencia orange on the rates of oxidation of D-limonene. The extract from the outer flavedo contained the highest antioxidant activity. The inner flavedo (the portion between the flavedo and

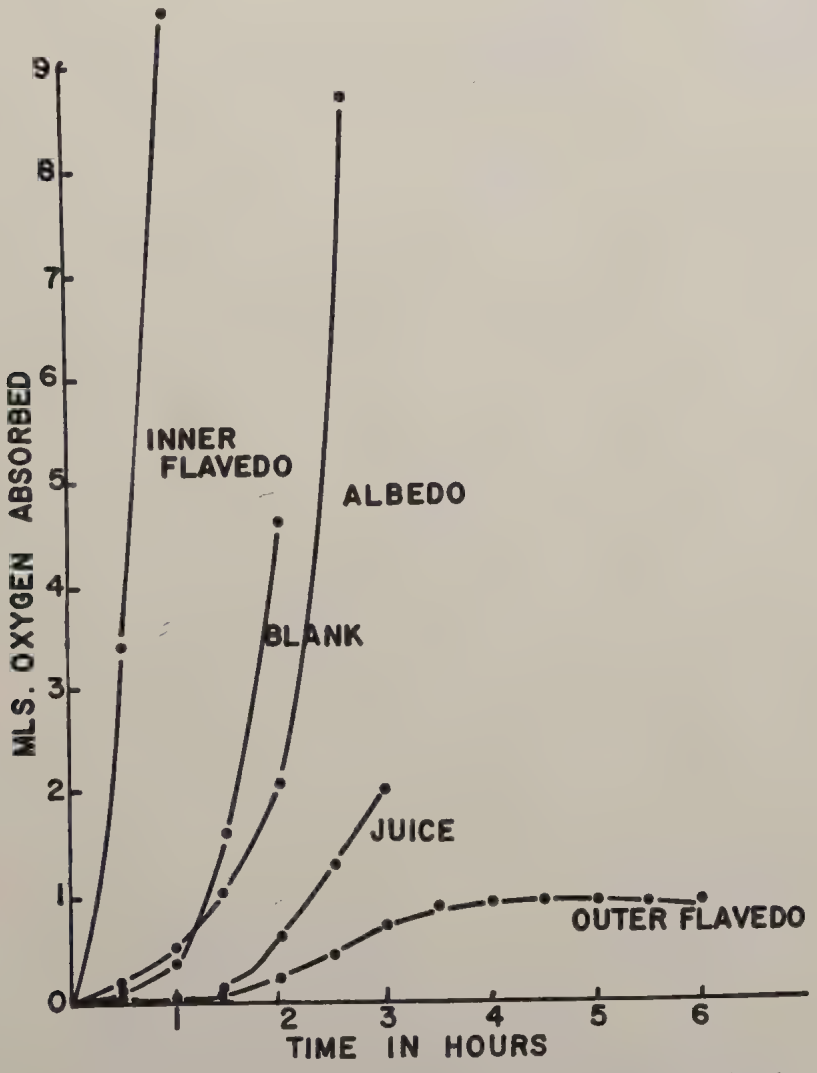


Fig. 78. Effects of extracts of component parts of Valencia orange on the rates of oxidation of D-limonene at 70° C. (158° F.). Extracts used: 0.5 gram of outer flavedo, 5 grams of inner flavedo and albedo, and 10 grams of juice. (From Ting and Newhall, 1965.)

albedo) accelerated the oxygen uptake of D-limonene, but the extract from the albedo had an activity not significantly different from that of the blank. The slope of the juice curve indicates that the juice contained sufficient antioxidant activity to produce a slight increase in the induction period of D-limonene.

Ting and Newhall (1965) also obtained some interesting data by correlating the antioxidant activity of extracts from the flavedo tissues of several citrus types with the autoxidation of D-limonene (figure 79). The lime and lemon flavedo extracts had approximately the same activities as the blank determination. This means that the induction period had an exceptionally

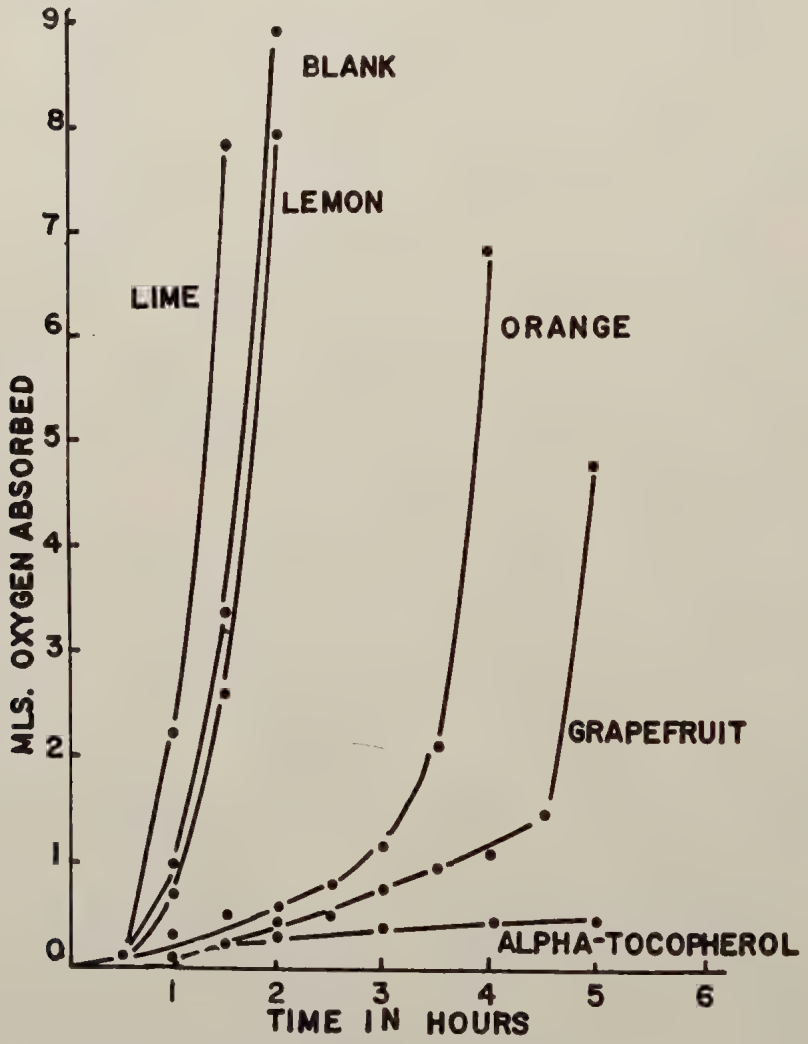


Fig. 79. Effects of extracts of flavedo of several citrus fruits on the rates of oxidation of D-limonene at 70° C. (158° F.). Extracts used were equivalent to 0.5 gram of fresh orange flavedo and 1 gram of the flavedos of the other fruits. The effects of 10 ppm of *alpha*-tocopherol are also shown. (From Ting and Newhall, 1965.)

short duration before the period of active oxygen absorption was initiated. The extracts from both orange and grapefruit flavedo significantly increased the induction period of the D-limonene samples. The curve for orange flavedo showed a shorter induction period than that of grapefruit because 0.5 gram of fresh orange flavedo was used in this instance instead of the 1 gram of flavedo tissue used in all other fruit experiments. For comparative purposes, the antioxidant activity of α-tocopherol, determined at 10 ppm, showed complete inhibition of the autoxidation of D-limonene.

The same investigators also noted that orange oil had antioxidant activity. After removing the volatile constituents from orange oil by vacuum distillation, the residue, free of the precipitated wax, was dissolved in petroleum ether.

Experiments were then performed with aliquot portions of this extract equivalent to 0.2 and 1 gram of the original oil. The results showed that the antioxidant activity of 1 gram of oil had slightly less activity than that of 0.5 gram of flavedo.

Wax in the Peel. Nelson and Mottern (1934), Markley *et al.* (1937), and probably others who determined the composition of cold-pressed grapefruit peel oil, found about 7 to 8 per cent of it to be composed of nonvolatile wax or waxlike substances. Markley and co-workers found that the nonvolatile waxy residue remaining after distillation of Florida grapefruit peel contained solid fatty acids of a mean molecular weight corresponding to $C_{32}H_{64}O_2$; linolenic, linoleic, and oleic acids; a saprogenic ketone, $C_{30}H_{52}CO$; hydrocarbons, $C_{29}H_{60}$ and $C_{31}H_{64}$; a phytosterol, $C_{28}H_{42}OH$; and umbelliferone, $C_9H_6O_3$. These substances had their origins in the cuticle wax of the peel. This wax was dissolved by the essential oils of the peel during the pressing process.

If the grapefruit oil was placed in storage at a temperature of approximately $-10°$ C. (14° F.), a light-yellow, waxy material with a pronounced taste and odor was precipitated. The crude wax averaged about 8.7 per cent of the weight of the oil. This wax, that settled out at a low temperature, had a crystalline structure and was easily filtered from the oil. It could be separated from the residual oil by vacuum distillation.

The lipoidal components of the cuticle, or outer covering, of the fruit are formed in the underlying protoplasts, and are exuded through minute channels to the surface of the epidermal cells, where they oxidize, dry and form successive layers (Scott and Baker, 1947). This wax coating on the surface of citrus fruits consists of a layer of flat scales which is equal in area to the surface of the epidermal cells. On the surface of maturing fruit, the thickness of the wax coating varies considerably. Overlapping of the scales was said by these authors to be owing to the continuing cell division and consequent expansion of the epidermis.

Naringin in Peel and Rag

In edible portions of the grapefruit, interest in naringin is centered upon keeping the content low enough to avoid undersirable bitterness. (See chapter 2, under the heading "Naringin," starting on page 133.) In the peel and in cannery waste, on the other hand, it is of interest as a marketable by-product (see chapter 6, page 543), and considerable work has been done on methods of extraction and analysis, as well as on the amounts present in the peel of different varieties or of fruit grown under different conditions. Some chemical characteristics are given in chapter 2, where just noted, and the structural formula is shown in figure 31 of that chapter. For further discussions of the flavonoids, see Baier (1955*a*, *b*) and Geissman and Hinreiner (1952).

Methods of Extraction. Zoller (1918*a*) appears to have been the first investigator in this country to devise a method for extracting naringin from grapefruit peel. The amounts he found in the peel of 3 varieties of grapefruit are shown in table 121. A comparison of the values for fresh and stored fruit in the last column will show that there was a noticeable decrease after periods of storage.

Poore (1934) extracted naringin from Marsh Seedless grapefruit peel and residue by applying the principle that it is very soluble in hot water and only slightly soluble in cold water. The finely ground residue was extracted with 4 parts of water for 5 minutes at 90° C. (194° F.). The residue was extracted a second time with 2 parts of water at 80° C. (176° F.), The extracts were combined, filtered, and evaporated to one-ninth of the original volume. The concentrate was then seeded with a few crystals of naringin, and placed in an ice box. Crystallization of naringin occurred in 2 or 3 days. The product could be purified by recrystallizing from hot water. Poore's data on California peel and rag ground together and on Florida peel and rag ground separately are given in table 87 of this chapter.

TABLE 121

The Naringin Content of the Peel of 3 Varieties of Florida Grapefruit.*

Varieties	Ages of fruit†	Weight of fruit (grams)	Quantity of naringin from peel only (grams)
Indian River	Fresh market	770	0.62
Indian River	Old market	715	.35
Walters	Fresh market	682	.50
Walters	Four months at 15° C. (59° F.)	695	.24
Marsh Seedless	Fresh market	539	.36
Marsh Seedless	Old market	570	.08

*From Zoller (1918*a*).

†It would be difficult to give the age to any great degree of exactness. "Old" fruit had been stored for some time to determine the decrease in amount of naringin.

Higby (1947*a*) was granted U. S. Patent No. 2,421,061 for the recovery of flavanone glycosides, in general, from plant-source material such as citrus pulp. The finely ground pulp material was mixed with a sufficient amount of calcium hydroxide to cause hardening of the material, and was then suspended in water. Sufficient alkali (sodium hydroxide or ammonia) was added to adjust the pH of the mixture to about 7.5 in order to solubilize the flavanone glycosides. The liquor containing the solubilized flavanone glycosides was acidified to below pH 6.0, and the precipitated glycosides were filtered from it.

Using similar procedures specific for naringin, Higby (1947*b*) received U. S. Patent No. 2,421,062 for a method for the recovery of naringin. The naringin-containing material, such as grapefruit pulp, was treated with calcium hydroxide to coagulate the various plant components. Sodium hydroxide was added to the lime-treated material in an amount sufficient to increase the alkalinity to between pH 8.4 and 9.1, in order to solubilize the naringin. This liquor was treated with hydrochloric acid to lower the acidity to pH 4.0 to 5.0. The naringin crystallized on standing.

Baier (1947) received U. S. Patent No. 2,421,063, similar to that of Higby, with the exception that the naringin was both extracted and solubilized with lime instead of using sodium hydroxide or ammonia to extract it. In other words, the Baier patent used the alkaline earth for both hardening and extraction of grapefruit pulp.

According to U. S. Patent No. 2,510,797 (Burdick and Maurer, 1950), naringin can be removed from grapefruit extracts and solutions by adjusting their acidity from pH 3.5 to 7.0 and then adding sufficient activated carbon to absorb the naringin. The mixture of the extract and carbon is then heated to approximately 60° C. (140° F.) and agitated, after which the carbon is separated from the extract.

Products of Hydrolysis and Treatment with Alkali. On hydrolysis with dilute acids, naringin yields naringenin, glucose, and rhamnose (Asahina and Inubuse, 1929; Poore, 1934). Asahina and Inubuse determined the yields of the products, and compared them with these theoretical values.

Percentages	*Theoretical*	*Found*
Naringenin ($C_{16}H_{14}O_6$)	44.1	43.4
Glucose ($C_6H_{12}O_6$)	29.2	31.9
Rhamnose ($C_6H_2O_2$)	26.6	22.8

Pulley and von Loesecke (1939) prepared rhamnose by hydrolyzing naringin with dilute sulfuric acid (3.7 per cent) to naringenin, glucose, and rhamnose. Their yield of rhamnose amounted to 20 per cent of the naringin taken, which was approximately 62 per cent of the theoretical amount. The amount of naringenin obtained was about 42 per cent of the naringin taken, or approximately 88 per cent of the theoretical value. The glucose was fer-

mented with baker's yeast, leaving the rhamnose intact in solution. They suggested that naringin recovered from canning-plant refuse could be used as a source material for the preparation of rhamnose.

Poore (1934) stated that when boiled with potassium hydroxide, naringin would yield phloroglucin and p-coumaric acid.

Color Reactions. Willstatter (1914) found that distinctive color reactions occurred when natural plant pigments were reduced in alcoholic solution with hydrochloric acid and magnesium. This so-called "cyanidin" reaction was used by Szent-Gyorgyi (1938) in studying the color reactions of bioflavonoids of citrus fruits; both the glycoside and the aglucone formed compounds which ranged in color from orange to purple.

According to Bryant (1950), the aglucone form, naringenin, may be distinguished easily from the glycoside by adding octyl alcohol to the "cyanidin" reaction mixture. In the isolation and purification of these bioflavonoids, it is possible with this test not only to distinguish between the glycoside and the aglucone, but also to determine when the hydrolysis of the glycoside to the aglucone form is complete.

The flavanone naringin gives a positive "cyanidin" reaction, as do hesperidin, quercitrin, rutin, and eriodictyol glycosides, but naringin does not give the Wilson (1939) boric acid color reaction, which is positive for the flavonol glycosides quercitrin and rutin. As observed by Wilson, a brilliant yellow color was produced when lemon juice was dried in the presence of boric acid. It was further shown that this coloration was produced by a reaction between the boric acid and a flavone or group of flavones similar in structure to quercetin.

If naringenin and hesperetin are treated with sodium hydroxide, a substance is formed that gives a positive test with the borocitric acid reagent. By converting the naringenin and hesperetin to their hydroxychalcone forms, a chemical configuration is produced that gives a positive test with the borocitric acid reagent. According to Wilson (1939), the flavanones gave no color reaction with boric acid, but all flavones except fisetin gave positive tests (figure 80).

In their search for a colorimetric method for the determination of naringin, Harvey and Rygg (1936*a*) noted that a vinaceous red color was produced when ferric chloride reacted with naringin at very dilute concentrations. In addition to the instability of the color at low concentrations of the reactants, the procedure was not highly specific for naringin, since other substances, such as hydroxy compounds and citric acid, interfered with color development. The reaction of ferric chloride with increasing concentrations of naringin produced darker colors which gradually developed into black.

Kesterson and Hendrickson (1957) used these various color intensities to show, morphologically, the presence of naringin in grapefruit tissues (figure

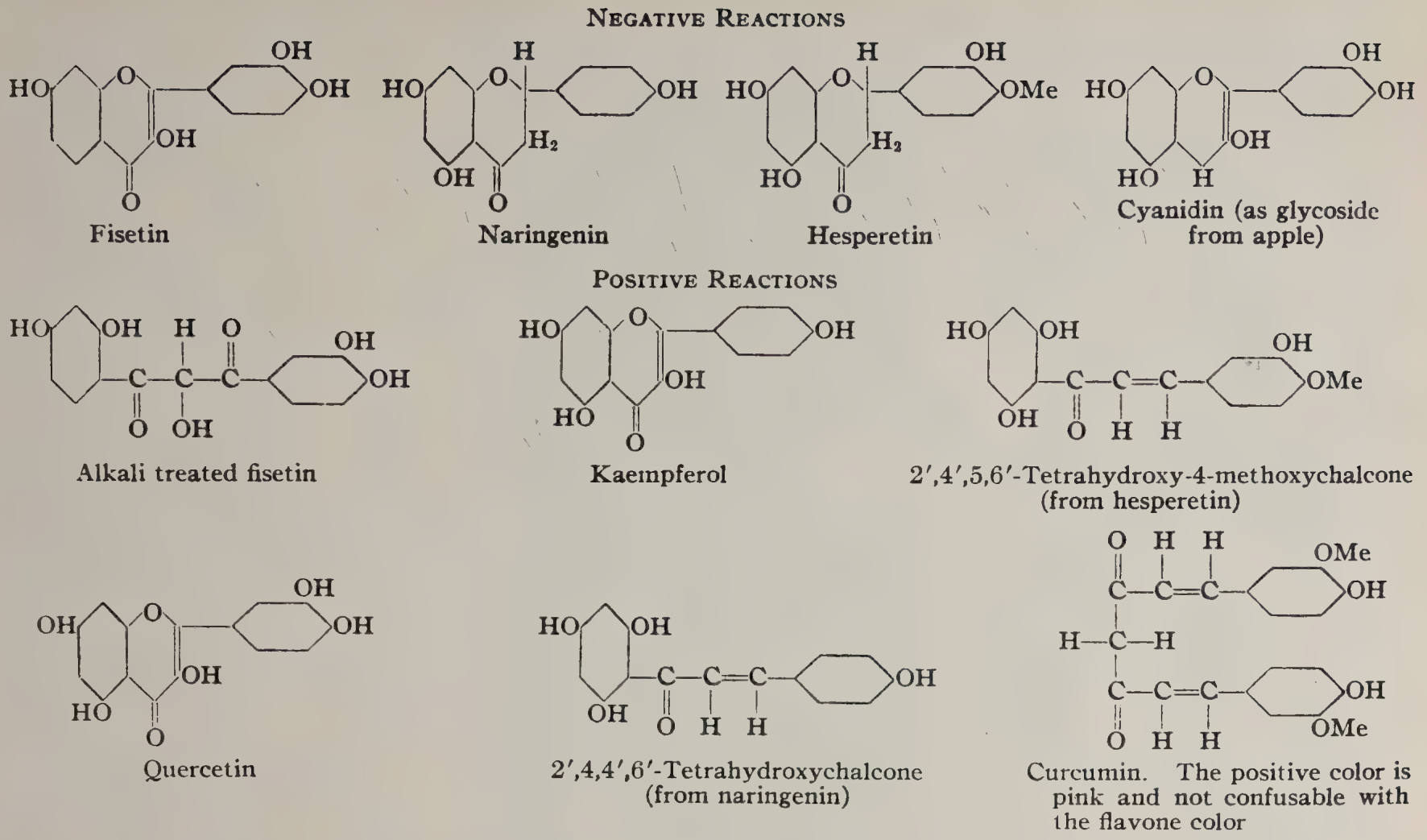


Fig. 80. Configurations of various organic substances, and correlation with boric acid reaction. (From Wilson, 1939.)

Fig. 81. Grapefruit sections, showing the location of naringin by adding ferric chloride to the cut surfaces. (From Kesterson and Hendrickson, 1957.)

81). This method of identification of naringin in the cut surfaces of grapefruit showed clearly the qualitative differences in the amounts of naringin in the various tissues.

Hendrickson *et al.* (1958*b*) developed an ultraviolet absorption method for determining the naringin content of grapefruit juices. After evaluating the important effects of solvents, dilution of samples, pH, and juice constituents on the accuracy of the procedure, they found that the last-named method gave results which compared favorably with the naringin values determined by the Davis method.

Occurrence and Amounts. In 1 lot of California grapefruit, Poore (1934) found the following percentages of naringin in different parts of the fruit: in juice, 0.06; in rag, 0.15; in whole peel, 0.90; and in albedo, 1.49. In 5 other

tests where the peel and rag had been ground together, he found 0.47, 0.49, 0.69, 0.90, and 1.08 per cent naringin. In separate tests of peel and rag taken from a Florida packing house, he found an average of 0.40 per cent naringin in the peel and 0.10 per cent in the rag, as shown in table 87 of this chapter.

Harvey and Rygg (1936*c*) found the naringin gradients to be relatively small in percentages of wet weight, as shown by the following values. (The averages result from several figures, and not precisely from the extremes shown here.) The areas given are in southern California.

Corona-Fontana area (between desert and coastal)
Peel, stem end ..... 4.62 to 8.31, averaging 7.31.
Peel, stylar end ..... 5.51 to 8.36, averaging 7.32.
Oasis area (desert)
Peel, stem end ..... 4.68 to 9.84, averaging 6.76.
Peel, stylar end ..... 4.69 to 8.57, averaging 6.17.

Sixteen determinations made by Harvey and Rygg (1936*a*) showed that the peel from Marsh grapefruit grown in the Corona-Fontana area averaged 8.07 per cent naringin content on March 19 and 5.07 per cent on September 6, while that of fruit from the Oasis area contained 9.21 per cent on November 6 and 5.26 per cent the following April 9. These concentrations are shown here, as percentages of wet weight in different parts of the peel.

Corona-Fontana area (between desert and coastal)
Flavedo ..... 4.17 to 7.87, averaging 6.38.
Albedo ..... 5.96 to 10.35, averaging 8.26.
Oasis area (desert)
Flavedo ..... 3.01 to 8.24, averaging 4.80.
Albedo ..... 6.85 to 10.17, averaging 8.15.

The naringin values of Harvey and Rygg (1936*a*) were determined by their method, which was based upon the color developed by the reaction of naringin with ferric chloride.

Rygg and Harvey (1938) verified their earlier suggestion (Harvey and Rygg, 1936*a*) that the changes in naringin content of grapefruit are controlled by factors that are poorly understood, and that the content might increase or decrease in storage according to the condition of the fruit as well as to the storage conditions. Their table 122 plainly indicates a tendency for the peel content of naringin to increase in storage through a wide range of temperatures, with an increasing trend as the temperatures rise. These authors also suggested that naringin might be an indirect product of respiration, since its amount was highest in the fruits exposed to the highest temperatures, in field and storage. Storage time for the fruit in table 122 was 6 weeks.

Davis (1947), as mentioned on page 135 of chapter 2, reported these amounts of naringin, in grams per 100 grams: juice vesicles, 0.140; segment membrane, 1.250; albedo, 2.100; flavedo, 0.600; and core, 2.600.

Maurer *et al.* (1950) reported the naringin contents of various tissues of 6 grapefruit varieties grown in Texas (see table 67, chapter 2). In all varieties, naringin was highest in the albedo and lowest in the juice. They also pointed out that between October 13 and November 17, 1948, the naringin concentration decreased 66 per cent in the flavedo, 45 per cent in the albedo, 53 per cent in the membrane, and 54 per cent in the core.

Kesterson and Hendrickson (1952, 1957) studied the monthly variations in the absolute amounts of naringin per fruit and in its percentage distribution among the fruit tissues. Their results are shown in table 68 of chapter 2 and in figure 81 of this chapter. The albedo and the "rag and pulp" (presumably after juice extraction) together contained about 90 per cent of the naringin.

A Second Glycoside from Pulp. Ma and Schaffer (1953) isolated a second glycoside, β-sitosteryl D-glycoside, from commercially dried grapefruit pulp. They obtained 8.65 grams of this compound from 150 kilograms (about 330 pounds) of dried pulp. The β-sitosteryl D-glycoside melted at 290 to 291° C. (about 554° F.). It was insoluble in water, alkali, acid, and most organic solvents, but was slightly soluble in methyl and ethyl alcohol and highly soluble in pyrimidine. They isolated β-sitosterol from grapefruit seeds in greater amounts than β-sitosteryl D-glycoside, which apparently existed only in trace amounts in the seeds.

Enzymes in Peel and Seeds

THE ENZYME GLYCOSIDASE was isolated by D. H. Hall (1938) from grapefruit leaves and from the tissues immediately under the flavedo. This enzyme hydrolyzed naringin *in vitro* at 37° C. (99° F.) and pH 7.0. He found the products of the reaction to be the insoluble aglycone naringenin (or naringetol) in crystalline aggregates belonging to the hexagonal system, and a new disaccharide sugar, distinct from but akin to rutinose. The aglycone linkage appeared to be through rhamnose instead of glucose. As far as can be found in the literature, this was the first time naringin had been hydrolyzed by an enzyme *in vitro.*

Phosphatase. Axelrod (1947) found the following PNP units of phosphatase in grapefruit peel tissues and seeds: 186 PNP in the flavedo press juice, 141 in the peel brei (mash), 113 in the filtrate from peel brei, 160 in the albedo brei, 127 in the filtrate from albedo brei, and only about 2 in the seeds. (One PNP unit is that quantity of enzyme that will liberate 1 γ of nitrophenol per minute per 7 milliliters of reaction mixture at 37.8° C. (100° F.)

Peroxidase. Davis (1942) reported the following amounts of peroxidase in the peel and seed tissues of Marsh grapefruit, in peroxidase units (PU) per kilogram of fresh tissue: 13.8 in the albedo, 350 to 414 in the outer flavedo

TABLE 122

NARINGIN CONTENT OF STEM END OF ALBEDO IN FRESH AND STORED GRAPEFRUIT FROM OASIS, CORONA, AND WOODLAKE, CALIFORNIA.*

Localities and bases	Not stored (per cent)	Percentages of naringin after storage (at degrees F. and C.)			
		68 (20)	56 (13.3)	48 (9)	38 (3.3)
Oasis: Dry-weight basis	10.25	.....	12.17	11.85	11.26
Fresh-weight basis	2.36	.....	2.71	2.60	2.52
Corona: Dry-weight basis	10.44	12.10	11.88	11.40	11.19
Fresh-weight basis	2.05	2.31	2.34	2.24	2.22
Woodlake: Dry-weight basis	8.03	.....	9.60	8.94	8.74
Fresh-weight basis	1.64	.....	1.90	1.76	1.70

*From Rygg and Harvey (1938).

TABLE 123

MAXIMUM PEROXIDASE ACTIVITY IN DIFFERENT CITRUS TISSUES.*†

Tissues	Varieties	Activity with:‡			
		Pyrogallol	Guaiacol	Metol	p-Phenyl-enediamine
Fruit peel (flavedo)	Shamouti orange	68 (12:7)§	100 (12:53)	174 (4:3)	250 (11:17)
	Sour orange	158 (9:3)	491 (11:110)	...	...
	Marsh Seedless grapefruit	158 (12:11)	509 (12:53)	...	...
	Lemon	360 (12:7)	1,455 (12:80)	...	...
Mature leaves	Shamouti orange	248 (12:7)	191 (8:53)	419 (7:3)	1,256 (370:17)
	Marsh Seedless grapefruit	370 (12:14)	955 (8:53)	...	...
	Lemon	375 (12:7)	709 (12:21)	...	...
Roots	Sour orange	1,000 (12:11)	2,625 (6:21)	...	...
	Sweet lime	1,267 (3:7)	5,000 (1:21)	2,351 (7:3)	2,200 (73:17)

*From Goren and Goldschmidt (1966).
†Tested at the optimum conditions for each H-donor separately, calculated from several sets of experiments involving different concentrations of H-donors and H_2O_2.
‡Absorptivity $\times 10^3$ in 120 seconds per milligram, fresh weight.
§The figures in parentheses give the concentrations of H_2O_2 and H-donor giving this maximum activity (both in millimoles).

(no test made on inner flavedo), 147 in the whole seeds, 440 to 1,100 in the inner seed coats, 5.5 in the outer seed coats, and 27.6 in the cotyledons. Other tests confirmed that peroxidase was especially abundant in the inner seed coats.

This was found to be particularly true for a seedy grapefruit (1,436 PU per kilogram) and, among other citrus fruits tested, the tangerine (1,100 to 2,500 PU per kilogram).

In studying the characteristics of peroxidase activity in various tissues and types of citrus, Goren and Goldschmidt (1966) observed that different tissues produced different activity values when peroxidase was determined by means of different H-donors (table 123). The root tissues were outstanding because they had significantly higher peroxidase activities with all donors used than the other tissues tested. The flavedo of Marsh Seedless grapefruit had a peroxidase activity halfway between the values reported for Shamouti orange and lemon. The mature leaves of the grapefruit had more than twice as much peroxidase activity as the flavedo. These authors noted that differences between H-donors could be easily observed when duplicate values were obtained on the same tissue, but with tissue samples of widely different peroxidase activity, the differences attributed to H-donors were small in comparison with those attributed to tissue characteristics. In all citrus types and their tissues, there was an interrelationship between the optimum concentration of H-donors and H_2O_2 for each tissue.

The data developed by Goren and Goldschmidt and reported in table 123 show the comparative peroxidase activities of tissues, determined under optimum conditions and at constant pH and temperature. These revealed the characteristics of each tissue at its maximum enzymatic activity.

Monselise and Kahan (1966) reported on the changes in composition and enzymatic activities of flavedo and juice of Shamouti orange following gamma radiation. In addition to a marked decrease in the ascorbic acid content of the flavedo, they noted an increase in peroxidase and a decrease in the catalase activities of the flavedo. The time course of changes in peroxidase activity of the flavedo showed differences from the controls within 2 days, but maximum effects were observed 8 days after irradiation.

External pitting and damage were much more pronounced on green than on mature fruit, the damage also being related to the irradiation doses as well as to the time elapsed between picking and radiation treatments. No damage was observed on mature fruit that was irradiated 12 days after picking with doses up to 168 krad. With mature fruit, the peroxidase activity was not significantly affected at any doses used on samples 12 days after picking. It thus developed that correlation coefficients between external fruit damage and peroxidase activity were highly significant for both green and mature fruit (+ 0.814 and + 0.802, respectively).

Acetyl Esterase. Jansen *et al.* (1947) isolated from grapefruit, orange, and lemon tissues an enzyme that hydrolyzed esters of acetic acid. This enzyme was labeled "acetyl esterase," since it was shown to be a typical esterase having its greatest activities on esters of acetic acid. The major portion of acetyl esterase occurred in navel flavedo and decreased toward the center of the fruit.

The rates of hydrolysis of the esters of acetic acid were from 0.2 to 0.4 of that observed on triacetin. Methyl and ethyl butyrates were hydrolyzed at a rate of 0.1 to 0.2 of that observed on triacetin. Tripropionin and ethylene glycol diformate were hydrolyzed at rates of 0.3 and 0.5 of that for triacetin. The relative rate of hydrolysis of tributyrin was exceedingly slow (0.04). Attention was drawn to the fact that acetyl esterase was not a lipase enzyme. The rate of hydrolysis of tributyrin was only 4 per cent of that of triacetin. Furthermore, acetyl esterase showed no detectable hydrolysis of olive oil. The pH optima for the action of grapefruit acetyl esterase on mono-, di-, and triacetin occurred between pH 5.5 and 6.5. The enzyme was inactivated at 35° C. (95° F.) and pH 7.0, and inactivation occurred at a more accelerated rate at 50° C. (122° F.).

Vines and Metcalf (1967), in studying the organic acid metabolism of the juice vesicles of Marsh grapefruit, isolated active mitochondrial preparations from the fruit during seasonal growth and development, and found this material to be capable of oxidizing intermediates of the citric acid cycle. Seasonal differences in mitochondrial activity occurred in grapefruit; activity increased from low to maximum values as the fruit approached maturity, and the trend declined with its further maturation (figure 82). The time course of changes in mitochondrial activity corresponded to similar changes in total acidity of the juice. This particular period in the growth cycle of the fruit is the most metabolically active one; consequently, changes in concentrations of chemical constituents occur in the tissues at an exceedingly rapid rate.

(See also, Metcalf and Vines, 1965.)

As illustrated in figure 82, succinate was the only substrate that possessed significant mitochondrial activity with preparations from immature fruit. The maximum rates of oxidation and phosphorylation occurred in July and early August. However, there was a delay in the ability of the mitochondrial preparations to oxidize α-ketoglutarate, as well as a delay in coupling α-ketoglutarate oxidation to phosphorylation. The P/O ratios increased progressively to maximum values in the January samples, and a rapid reduction followed immediately. The succinate experiments were exceptions; the P/O ratios reached a maximum of 1.3 in September, and gradually declined during the winter months. Soon after the cold weather in mid-January, the phosphorylative reactions increased with each substrate, except malate.

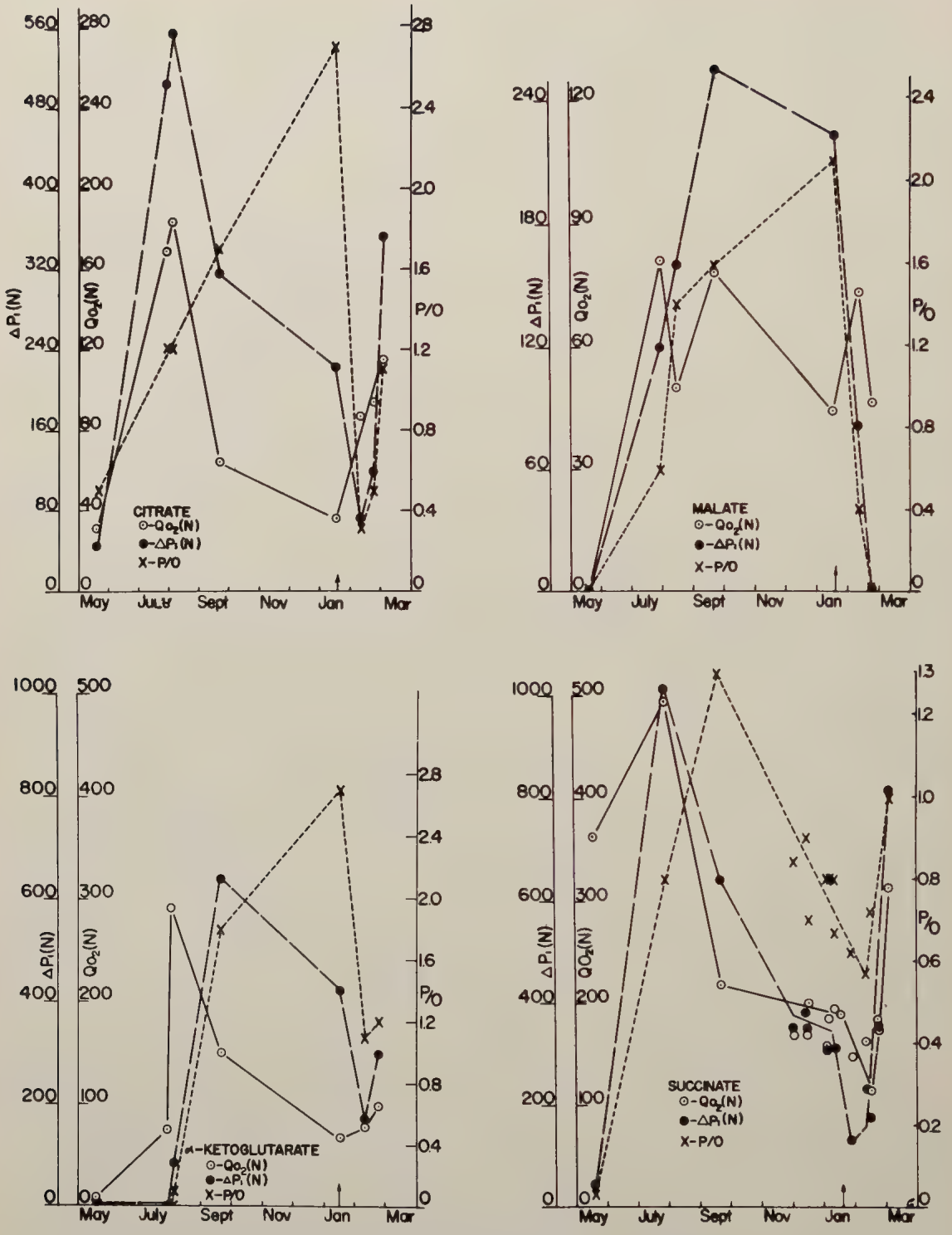


Fig. 82. Seasonal changes in respiration and oxidative phosphorylation of mitochondrial preparations from grapefruit. $Qo_2(N) = \mu^1/O_2$/mg N/hr.; $\Delta P_i = \mu$ g P/mg N/hr. (From Vines and Metcalf, 1967.)

Peel Pigments and Degreening

PHLOBATININ AND CAROTENOIDS were studied by Hardy and Warneford (1925), who found the yellow coloring matter in the peel of limes, lemons, and grapefruit to be phlobatinin in solution in the cell sap, rather than carotenoids in the plastids. The results obtained in the meantime appear to be somewhat contradictory, and need further study.

Miller (1938) found carotenoids in limes, lemons, and grapefruit that were mature, but had peels that were still green in color. (For a discussion of carotenoids, see chapter 2, starting on page 183.) However, the green-skinned fruits lost most of their carotenoids when they were artificially degreened with ethylene, or when they normally degreened on the tree. This was the reverse of what took place with oranges and tangerines; in these, the carotenoids increased as degreening occurred. Miller presented the following data on the carotenoid content of the peel of mature Duncan grapefruit, stated as milligrams per 100 grams.

LOT 1	*Carotene*	*Xanthophyll*
Mature, green	0.150	1.040
Degreened with ethylene	0.075	0.510
LOT 2		
Mature, green	0.045	0.435
Degreened on trees (full-ripe color)	0.005	0.000

The second lot was normally much paler in color; note the differences in the amounts of carotenoids. A later report by Miller and Winston (1939) gave similar data. These results appeared to confirm the findings of Hardy and Warneford (1925), mentioned above, that the yellow color in grapefruit was caused by phlobatinin rather than carotenoids.

French and Abbott (1948) did not find any carotene in grapefruit peel. Although several of the carotenoid pigments are considered to be precursors of vitamin A, it should be noted that grapefruit refuse, including the peel, is almost totally lacking in these precursor pigments.

Natural and Ethylene Degreening. Most of the studies on degreening have been made on oranges or noncitrus fruits; therefore, the chemical and other changes that occur in degreening will not be reviewed here in detail.

Thornton (1940) pointed out that degreening might occur on the trees, in storage, or during treatment with ethylene, and that the last-named process did not constitute "artificial ripening." Ethylene merely accelerates the natural processes which normally destroy the green color (chlorophyll).

Noack (1943) proposed that the first step in degreening should be the proteolysis of the plastid stroma. It thus seems that degreening is hastened because the ethylene accelerates the activity of the proteolytic enzymes. This appears to have been confirmed by the cytological observations of DuFrenoy

(1929). He noted a "fatty degeneration" of the chloroplasts with the formation of oil drops in which the yellow pigments dissolved.

Roper and Miller (1951), in summarizing results of earlier work in this field, stated that degreening of the fruit occurs because (1) ethylene and enzymes hydrolyze the plastid stroma, and thus make available materials that can be used in respiration; and (2) this leaves the chlorophylls unprotected, hence they are acted upon by chlorophyllase and are oxidized by hydrogen peroxide in the presence of a ferrous hydroxide catalyst, or by a ferric hydroxide plus a cuprous hydroxide catalyst. The general conclusion drawn is that ethylene merely accelerates the normal degreening processes.

Fiber in Peel and Rag

THE CRUDE FIBER content of fresh grapefruit peel and rag was checked by Poore (1934). He ground fresh California grapefruit peel and rag together, and found 2 per cent fiber. He also tested peel and rag separately from sterilized and stored Florida grapefruit, and found 1.71 per cent fiber in the peel and 1.44 per cent fiber in the rag on a fresh-weight basis. Poore noted that the California and Florida figures were not comparable because of the differences in freshness and moisture content. (See table 87 of this chapter.)

Chapter 4

Factors Affecting Quality, Flavor, Yield, and Maturity

MANY FACTORS affect the chemical and physical composition of the grapefruit, as the foregoing chapters have indicated. Out of the extensive research done along this line, there emerge certain basic conditions that exert a large influence on quality, flavor, yield, and maturity.

The quality of grapefruit, as well as the quantity, is the ultimate result of fruit setting, growth, development (tissue differentiation), and ripening of the fruit on the tree. In this developmental process, there occurs a complex of factors, internal and external, which produces various normal life-cycle changes. Some of these are so apparent as to be matters of common knowledge, while others are so obscure as to require the application of highly specialized techniques in order to demonstrate their existence. The nature of fruit growth consists of an integrated sum of the growth processes of individual tissues and cells. From a horticultural standpoint, the most conspicuous growth processes are used to determine the seasonal rates of growth as well as the quality of the fruit at harvest time and under postharvest storage conditions. They include, among many other considerations: (1) increase in fruit size and juice content; (2) development of adequate external color; (3) firmness of the flesh; (4) changes in total soluble solids, total sugar content, and total acid concentration in the juice; (5) ratio of total soluble solids to total acids; and (6) seasonal changes in coloring constituents in the pigmented grapefruit varieties.

The chemical and physical composition of the grapefruit thus affects fruit quality. The fruit must be mature and of good eating quality when harvested. It should be noted that so long as the fruit remains on the tree, its composition is changing, both physically and chemically.

Quality Defined. Wood and Reed (1936, 1938), in Texas, determined factors that affected fruit maturity and quality. They stated that grapefruit of good quality should have the following characteristics: (1) a relatively thin peel; (2) regular segments; (3) a large volume of juice; (4) tender flesh; (5) absence of bitterness; and (6) a ratio of soluble solids and acids that would give a tart, sweet taste.

Kebby and Skepper (1948), in New South Wales, defined good quality as being of normal shape and appearance common to the variety in question,

sound, smooth, thin-peeled, fine-textured with a minimum of rag, juicy, right in flavor, even in color, and free of surface blemishes, pests, and diseases.

Cowart and Stearns (1941) pointed out that at first, the idea was to get the fruit to market, even though it was not yet of maximum quality. Later on, a demand developed for grades based on internal quality. This called for the determination of the effects of such factors as fertilizers, rootstocks, and chemical sprays.

The attributes of quality just mentioned are stated and viewed in terms of the market acceptability of fresh fruit. These are factors that are evaluated by the human senses of sight, taste, touch, and smell. At the federal level of regulatory authority, the word "fresh" has been delineated and distinguished between fresh fruits and vegetables and processed fruits and vegetables. The processed product has been defined as "any fruit, vegetable, or other food product which has been preserved by recognized commercial process, including, but not limited to, canning, freezing, dehydrating, drying, the addition of chemical substances, or by fermentation." Consequently, the definition of the term "fresh," as applied to fruits and vegetables, is properly applicable only to products which remain in their natural state and which have not been processed in the manner described in the quoted definition.

In the fresh-fruit market, the term "quality" has come to have many meanings in the marketing of citrus fruits. Common agreement can be reached that fruits must be fresh, properly stored, or preserved if they are to possess their respective desirable properties. Beyond these obvious requirements, differences begin to appear in regard to details of the physical and chemical properties that go to make up the sum total of the characteristics that enter into "quality." Differences in the tastes and desires of consumers are the compelling reasons that cause farmers and producers to be constantly on the lookout for newer and better varieties of fruits and vegetables. The important quality of *taste* influences the methods used in harvesting and handling citrus fruits.

The factor of color is associated with many characteristics of food quality. The consumer does not taste color or the substances that produce it, but he does associate color with many features, such as flavor, nutritive level, texture, and other characterististics which contribute to the wholesomeness and attractiveness of citrus fruits.

From a fundamental and economic standpoint, quality has come to mean to the grower the attributes of a product that affect the price a buyer is willing to pay. These characteristics may be physical, chemical, or psychological. In citrus fruits, size, freshness, and color may be factors that are concerned chiefly with habits, preferences, and consumer attitudes. In the consumer market, physical appearance is the principal criterion of excellence in quality. Appearance is, indeed, an important factor in evaluating quality, but the extent

to which it should be used alone as an index is difficult to determine. It is known that the appearance of citrus fruits in the fresh-fruit market does have an important bearing on their selling price. Appearance embodies such features as color, shape, size, consistency, glossiness, and defects, all of which are important when judging quality.

1. *Texture and Consistency.* In the fresh-fruit market, structural properties are characteristics of prime importance. The satisfaction in eating fresh citrus fruit is derived from the structural as well as the chemical composition of the fruit itself. The consumer expects to squeeze juice from oranges and lemons. It must therefore be recognized that texture and consistency are markedly affected by the stage of maturity at harvest and by the environment in which the fruit has been grown.

2. *Flavor.* Flavor is one of the chief attributes of quality. In citrus fruits, most flavors are produced by aromatic substances, chiefly the volatile essential oils. It is well recognized that the primary sensations of taste are sweet, sour, bitter, and salty, but the number of flavors and aromas which can be derived from combinations of aromatic and volatile substances is infinite.

Until recently, it has been impossible to determine many of the volatile constituents of citrus fruit that affect flavor, because no analytical procedures were available for determining these important organic constituents that exist in the fruit in such small concentrations. This limitation has been overcome by the discovery of both paper and gas chromatographic methods for the qualitative and quantitative determination of chemical constituents of citrus fruits. By applying these analytical procedures, the accumulation of biochemical and physiological data has increased to the point where flavor factors can be accurately evaluated in relation to fruit quality.

It is a difficult problem to understand and evaluate flavors. The consuming public responds differently to flavor stimuli, as well as to differences in acuteness of taste. With respect to the sale of fresh citrus fruits, flavor is highly correlated with quality in the minds of prospective purchasers.

3. *Nutritive Properties.* These have been subjects of considerable investigation, and the results of the studies have contributed immensely to the quality factor of fresh citrus fruits. Nutritive value has come to play a prominent part in the consumer's idea of quality. Shoppers are seeking knowledge of nutrition on which to base their selection of foods. Food purchases are made with reference to vitamins and minerals. In the past, purchasers have made fruit selections on the basis of physical appearance and flavor rather than that of nutritive value. With the advance of knowledge in human nutrition, it can be expected that the consumer will pay increasing attention to the nutritive values of all foods on the fresh-fruit market.

It is well known that enormous differences exist in the composition of citrus fruits, as influenced by factors such as those that follow.

a) *The stage of maturity* at which citrus fruit is picked markedly affects the total soluble solids and volatile constituents that go to make up its total composition. These variations in composition are important considerations in maturity, especially in early-season harvests when prices of fresh fruit are favorable to the grower.

b) *The handling, harvesting, and time* between harvesting and consumption by the purchaser affect the composition of the fruit and consequently its nutritive value.

Environmental factors affect the composition and quality of citrus fruits. The importance of temperature to fruit growth has been strikingly demonstrated. Grapefruit is outstanding in the difference in time required for the fruit to mature under different climatic conditions. Buds on the trees come to full bloom and set fruit at approximately the same time at Riverside and Brawley, California, but about 13 months are required to mature the fruit at Riverside and only about 7 to 8 months at Brawley. The shortening of the time required at Brawley is chiefly owing to the excessive heat units, which accelerate the reactions of growth and elaboration of food materials.

When citrus fruits undergo processing, the "quality" factor of the product takes on additional and often different characteristics from those of the fresh fruit. Changes that are produced which alter fresh-fruit characteristics also produce correlative changes in fruit quality. In the processing of citrus fruits, the total quality value of the product may be broken down and related to the commodity produced, thus allowing each characteristic to be measured and controlled independently. Therefore, it follows that many physical peculiarities of the fruit that are so essential to the fresh-fruit consumer are of secondary importance when fruit is diverted to processing channels.

In studying the effects of processing variables on the quality of grapefruit juice, Hendrickson *et al.* (1958*a*) used ultraviolet absorption techniques within the 260- to 360-millimicron range to demonstrate significant differences resulting from the effects of these variables. The variables that were found to produce changes in the ultraviolet absorption of grapefruit juice were extractor pressure, peel extractives, rag and pulp additions, length of storage at room temperatures, aeration, and the degree of heat stabilization. They concluded that the increase in a juice's ultraviolet absorption would seem to be proportional to the amount of abuse treatments given to it. The effects of certain abuses that influenced the ultraviolet spectra of the juice were determined on 3 aliquot portions which received the following treatments: (1) aeration for 5 minutes with a high-speed stirrer, (2) partial evaporation of sample and replacement of evaporated portion with pure water, and (3) allowing a portion to stand at room temperature for 20 hours. The abuse treatments were listed in the order of their effects on increased ultraviolet absorption. This procedure had a practical aspect in that it measured certain

processing factors that were highly correlated with flavor deterioration in grapefruit.

Grapefruit juices from different sources showed extreme variation in absorption in the ultraviolet. The commercial juices showed the most absorption, and the hand-extracted juices showed the least. The absorption curve for each concentrate had a prominent peak at 285 mμ, with a high and broader reading at 325 mμ. The pronounced peak at 285 mμ indicated the presence of a relatively high naringin content. The hand-extracted juices produced curves with small peaks at 285 mμ, and much less absorption at 325 mμ.

Factors Affecting Quality and Quantity of the Peel

THE THICKNESS of the peel changes during the growth and maturation of the fruit. The study of these changes appears to have been begun a little over 50 years ago. For example, López-Domínguez (1919), in Puerto Rico, reported that fresh, mature Triumph grapefruit had 28.8 per cent, Marsh 27.2 per cent,

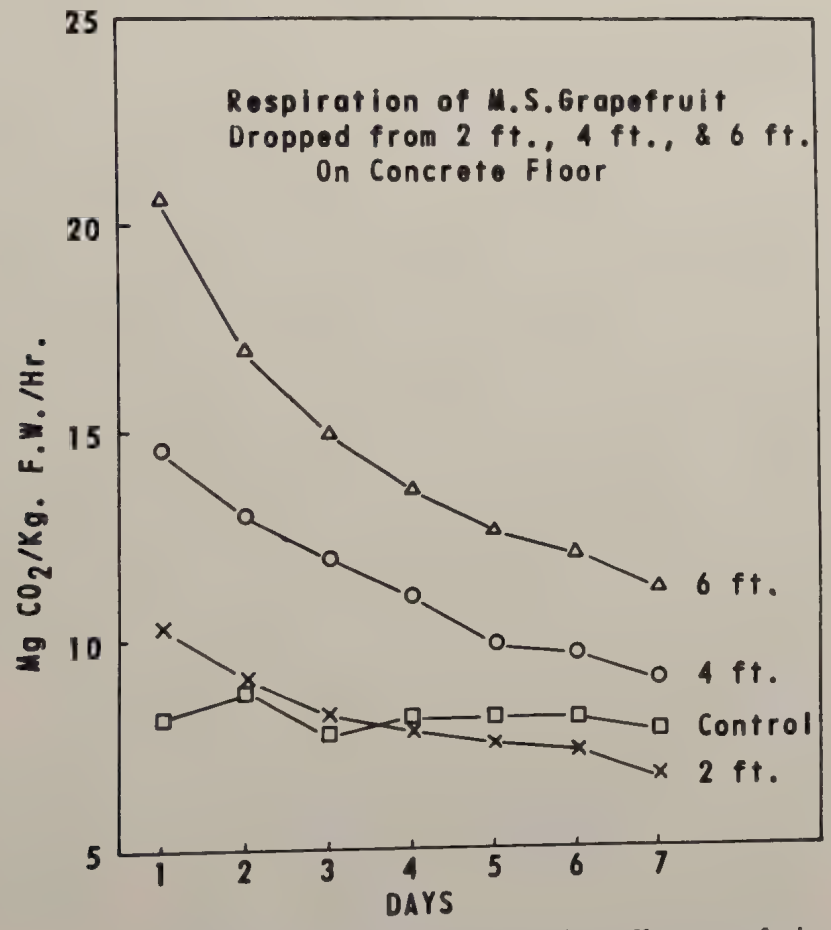


Fig. 83. Respiration rates of Marsh Seedless grapefruit when dropped from various heights onto a hard surface. (From Vines *et al.*, 1968.)

TABLE 124

PERCENTAGES OF PEEL IN MARSH GRAPEFRUIT GROWN IN DIFFERENT AREAS OF CALIFORNIA.*

Dates (1931–32)	Fruit weight (grams)	Peel (per cent by weight)
Average values for 8 localities in the Imperial Valley		
September 23	224	56.2
October 15	247	47.6
October 30	289	46.5
November 15	323	44.0
December 1	301	43.1
Average values for 5 localities in the Coachella Valley		
September 22	292	56.2
October 15	301	47.6
November 1	354	46.5
November 15	353	44.0
December 2	375	43.1
Average values for 17 localities in southern California		
February 16	267	45.4
March 2	257	45.0
March 16	277	44.5
March 30	277	44.1
April 15	281	44.2
May 1	289	44.4
May 18	300	42.8
June 1	300	42.8
Average values for 10 localities in central California		
October 13	222	52.0
November 3	231	49.2
November 17	258	47.4
December 3	256	46.7
December 16	259	44.9
January 1	259	47.0

*Adapted from R. H. Higby: Maturity work on citrus fruits. Sunkist Growers Research Department, File 15, Vol. 7:1–96. Unpublished data gathered in 1931–32.

and Duncan 28.3 per cent peel. Both percentage and thickness of peel decreased as the fruit matured, and then remained fairly constant.

Hawkins and Magness (1920) found that, on a fresh-weight basis, the amount of peel on the Florida common grapefruit ranged from 23.3 to 24.8 per cent, while its thickness ranged from 4.7 to 6.3 millimeters.

Physical Handling and Metabolic Activity. To show the effects of physical handling on metabolic activity, Vines *et al.* (1968) dropped Marsh Seedless grapefruit from various heights onto a hard surface, and determined the effects of these treatments on the respiration rates of the fruit (figure 83). Fruit that was dropped 2 feet (61 centimeters) had about the same respiration rate as the control sample, but fruit dropped 4 and 6 feet (122 and 183 centimeters) showed a significant initial increase in carbon dioxide production, which gradually receded toward the normal respiration rate over the 7-day test period.

Age of Fruit and Locality. Most of the available data record results based on both maturity and locality, so these 2 factors will have to be considered under the same heading.

Hawkins (1921) reported tests on Florida grapefruit in which, from July 29 to December 5, the thickness of the peel in 1 lot decreased from 6.8 to 4.5 millimeters, a reduction of 34 per cent; and in another lot, from 8.1 to 3.2 millimeters, a reduction of 60 per cent.

R. H. Higby (unpublished) determined the average percentages, by weight, of Marsh grapefruit peel in 4 different California areas at different times of the year. His results are shown in table 124, which also states the source reference. The data show that the relative weight of the peel decreased in all 4 areas as the season advanced and the fruit increased in size.

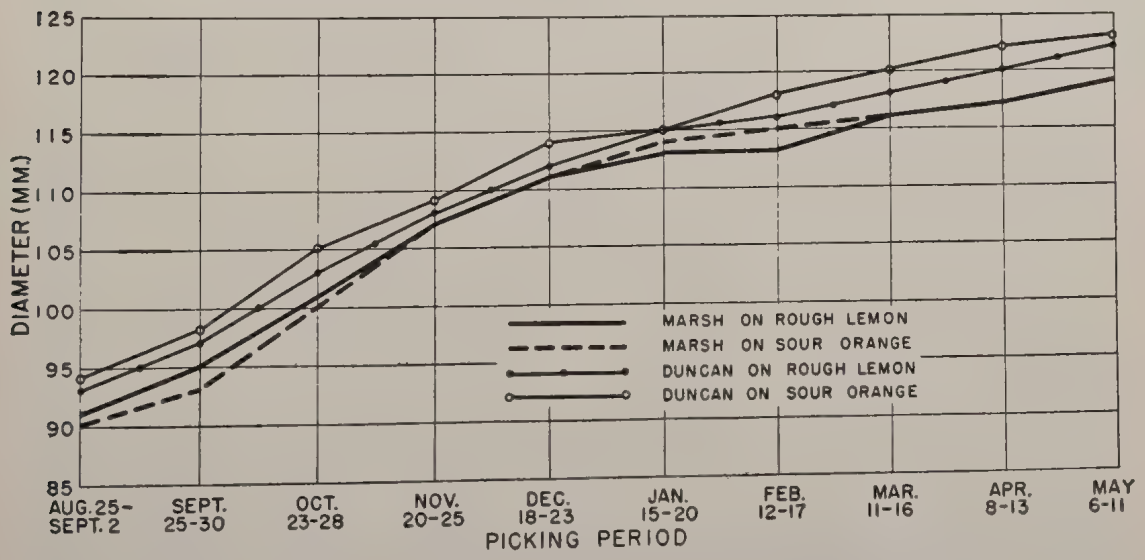


Fig. 84. Seasonal changes in the average diameters (in millimeters) of Marsh and Duncan grapefruit grown on 2 different rootstocks. (From Harding and Fisher, 1945.)

TABLE 125

SEASONAL CHANGES IN THICKNESS OF GRAPEFRUIT PEEL IN RELATION TO GROWTH OF FRUIT, SALT RIVER VALLEY, ARIZONA.*

(Average data, 60 fruits from a single grove each date.)

Dates (1939–40)	Thickness of peel (millimeters)	Peel (per cent)	Volume of fruit tested (cubic centimeters)	Growth of fruit (cubic centimeters)†
September 7...	10.7	46.0	320	301
22...	10.2	40.1	354	326
October 5.....	9.2	38.6	349	351
19.....	9.2	38.0	375	375
November 2...	8.9	36.5	399	395
16...	8.4	34.9	401	415
December 2....	9.0	36.0	434	440
13....	9.1	37.0	442	455
28....	9.2	36.8	472	476
January 18.....	9.0	37.6	476	493
February 6....	10.4	39.3	561	512
20....	10.4	39.7	558	542
March 5.....	10.5	40.8	541	571
19.....	10.5	42.3	549	596
April 2.....	11.1	43.4	624	614
16.....	10.2	42.2	604	616

*From Hilgeman (1941).

†Growth of 75 fruits from measurements by Harris. (Harris, Karl. Growth studies of Salt River Valley grapefruit. Unpublished data obtained in 1940.)

Seasonal changes in fruit size were determined by Harding and Fisher (1945) by measuring the diameters of the fruit during the growing period (figure 84). A constant increase in the diameter of the fruit occurred during its growth. The greatest increase occurred between August and January. After January, the increase in diameter was less rapid. When the diameter is expressed on a volume basis, the fruit increased in size about 15 per cent between mid-December and mid-March. The variety had a greater effect on fruit size than did the rootstock; Duncan was somewhat larger than Marsh.

Hardy and Rodríguez (1935) determined the percentages of peel in grapefruit from 2 estates in Trinidad, as shown here. (See table 58, chapter 2.)

EL QUEMADO (*"lime-land"*)		MAQUERIPE (*"acid-land"*)	
Choice fruits	22.9	Choice fruits	20.5
Cull fruits	23.8	Cull fruits	25.6

Hilgeman (1941) found that in the Salt River Valley and Yuma areas of Arizona, the percentage of peel, which was typically high in September, decreased rapidly to a low point in October and November. A gradual increase took place then, until a second high was attained in April and May. Hilgeman's conclusions appear to be substantiated by the data he presented. Tables 125 and 126 give his results on both peel thickness and percentage.

[*Turn to page 292.*]

TABLE 126

AVERAGE SEASONAL CHANGES IN PERCENTAGES OF ARIZONA GRAPEFRUIT PEEL, BY WEIGHT.*
(Highest and lowest values found on the day tested.)

Dates	Salt River Valley (12 groves)					Yuma (5 groves)				
	1937	1938	1939			1937	1938	1939		
			Av.	High	Low			Av.	High	Low
September 8	40.9	39.6	46.5	50.0	43.2	....	42.8	44.8	46.0	41.8
16	39.8	39.4	44.4	47.7	41.4	37.6	40.8	43.8	45.3	40.7
23	39.1	37.6	40.2	44.1	37.4	39.9	39.1	41.3	44.0	37.5
30	37.2	36.1	41.4	43.8	37.6	38.0	37.4	40.4	42.2	36.8
October 7	37.2	36.3	39.5	42.5	36.2	36.6	37.0	38.9	41.1	34.8
14	36.4	35.5	37.8	40.4	34.5	38.6	36.4	37.9	39.1	34.8
21	35.8	34.1	36.8	39.6	33.4	35.5	37.0	37.0	38.4	33.4
28	35.8	34.7	36.1	41.3	34.3	36.9	35.3	36.2	37.4	33.1
November 3	35.7	34.7	37.9	39.9	34.1	35.8	35.8	36.1	38.0	33.8
11	35.2	34.8	35.6	38.6	31.1	36.5	34.4	35.2	36.5	32.3
18	36.1	34.3	34.7	37.6	30.7	35.5	35.4	35.3	37.0	33.3
25	35.9	34.1	33.8	37.9	28.5	35.1	35.6	35.8	36.8	33.9
December 2	36.1	33.5	34.7	37.7	31.4	35.4	35.1	35.8	37.3	31.3
9	36.5	34.1	34.2	37.4	31.1	34.7	35.0	35.7	37.3	33.5
16	37.4	34.3	34.8	37.7	30.9	35.6	35.7	36.7	38.0	34.4
23	37.0	35.5	37.1	40.2	33.2	35.1	36.9	36.8	39.8	34.0
30	37.3	34.7	36.2	39.6	32.4	35.8	35.8	36.9	38.6	34.5
January 6	38.2	35.8	25.1	39.4	30.6	36.7	37.3	36.5	38.5	33.0
13	....	35.9	36.0	38.6	33.0	36.6	38.0	38.1	40.3	35.3
20	....	35.3	36.6	40.8	33.7	....	38.5	37.2	39.2	34.2
28	....	35.1	....	....	....	....	....	....	....	....
February 21	....	35.0	37.5	42.3	35.0	....	....	....	....	....
March 21	....	37.8	40.9	46.3	34.4	....	....	....	....	....
April 20	....	37.7	40.2	45.5	35.7	....	....	....	....	....
May 22	....	35.7	37.2	41.1	33.6	....	....	....	....	....

*From Hilgeman (1941).

TABLE 127

Effects of Various Factors on the Thickness of Florida Grapefruit Peel.*

Varieties and rootstocks	Localities	Seasons	Peel thickness (millimeters)†									
			Aug. 25	Sept. 25–30	Oct. 23–28	Nov. 20–25	Dec. 18–23	Jan. 15–20	Feb. 12–17	Mar. 11–16	Apr. 8–13	May 6–11
Marsh on rough lemon.....	Davenport	1939–40	10	10	8	9	9	11	11	11	11	11
		1940–41	8	6	6	7	7	7	6	8	7	8
		1941–42	8	7	7	7	8	8	8	8	8	8
Marsh on rough lemon.....	Lake Hamilton	1940–41	..	7	7	8	8	8	8	8	9	9
		1941–42	11	8	8	8	9	9	8	8	9	8
Marsh on rough lemon.....	Windermere	1939–40	9	8	9	9	9	8	9	8	8	9
		1940–41	8	7	6	7	7	7	7	6	8	8
Marsh on rough lemon.....	Bradenton	1939–40	8	7	6	6	6	8	7	8	8	8
		1940–41	8	6	6	5	6	5	5	6	6	6
		1941–42	7	6	5	5	5	6	5	6	6	7
Marsh on rough lemon.....	Fort Pierce	1939–40	8	7	7	7	7	8	8	8	8	8
		1940–41	7	6	6	6	6	6	6	7	7	7
		1941–42	7	6	6	6	6	6	5	5	6	7
Marsh on sour orange.....	Davenport	1939–40	10	8	8	7	8	..	..	..	..	..
		1940–41	8	7	7	7	8	7	7	8	8	8
		1941–42	8	6	6	6	7	7	7	7	8	8
Marsh on sour orange.....	Bradenton	1939–40	7	6	5	6	6	8	8	8	8	8
		1940–41	7	6	6	6	7	6	6	6	7	7
		1941–42	7	6	5	5	6	5	5	5	5	6
Marsh on sour orange.....	Vero Beach	1939–40	8	7	6	6	7	7	7	8	9	8
		1940–41	6	6	6	5	6	6	6	6	6	7
		1941–42	7	5	5	5	4	5	5	4	5	6
Marsh on sour orange.....	Homestead	1941–42	7	7	7	7	6	6	6	7	7	8

Duncan on rough lemon....	Davenport	1939–40	9	8	8	8	8	9	10	9	10	10
		1940–41	7	7	7	7	7	7	7	8	8	8
		1941–42	8	7	8	7	7	7	7	7	8	8
Duncan on rough lemon....	Lake Hamilton	1940–41	..	7	7	7	7	7	8	8	8	8
		1941–42	10	9	8	8	8	7	8	7	8	8
Duncan on rough lemon....	Palmetto	1939–40	7	6	6	7	7	8	7	8	8	8
		1940–41	7	6	6	6	6	6	6	7	7	8
		1941–42	7	6	5	5	6	6	6	6	6	7
Duncan on rough lemon....	Fort Pierce	1939–40	8	7	7	7	8	9	8	9	9	10
		1940–41	7	6	6	6	6	6	6	6	7	7
		1941–42	7	6	6	6	6	6	7	6	6	8
Duncan on rough lemon....	Homestead.....	1939–40	7	7	6	6	6	7	8	..	..	..
		1940–41	6	6	6	6	7	6	6	6	7	8
		1941–42	..	7	6	7	6	6	..	..	..	..
Duncan on sour orange....	Palmetto	1939–40	7	6	6	6	7	8	7	7	7	8
		1940–41	6	5	5	5	6	6	5	5	6	7
		1941–42	7	6	5	5	6	6	6	7	6	7
Duncan on sour orange....	Vero Beach	1939–40	6	6	6	6	7	8	7	8	8	7
		1940–41	7	6	5	5	6	6	6	6	7	7
		1941–42	7	5	5	6	6	5	5	5	6	8
Foster Pink on sour orange.	Bradenton	1939–40	7	7	6	5	6	7	7	7	..	..
		1940–41	6	5	5	5	5	5	5	6	6	6

*Adapted from Harding and Fisher (1945).
†Each value for peel thickness represents a mean of 25 determinations for 1 season.

Harding and Fisher (1945) made studies to determine the effects of various factors on the thickness of grapefruit peel. Their measurement data are shown in table 127, which has been adapted from several of their many tables on the composition of grapefruit. When checking the variations in peel thickness of Florida grapefruit, they found that it was usually 1 to 2 millimeters thicker during immaturity and senescence than it was when the fruit was in prime eating condition. They also found some indications of seasonal behavior, since the average thickness of the peel was slightly greater during the 1939–40 season than in the 2 following ones.

Fertilizers. Certain fertilizers appear to have an effect on the texture and thickness of the peel, while others do not.

Chapman *et al.* (1943) found that none of the fruits having a high nitrogen content showed any change in peel texture.

Jones *et al.* (1944*a*), in Arizona, reported that grapefruit from trees having a high nitrogen content throughout the year had thicker peel and poorer shape than those from trees having a declining nitrogen content during growth and maturation.

Innes (1946) found that the peel thickness of grapefruit was increased by the application of potassium, but decreased with the application of superphosphate in phosphate-deficient soils.

Rootstock and Soil Types. Harding and Fisher (1945) showed that when the grapefruit trees were on rough-lemon rootstock, and especially when grown on light, sandy soils of the ridge district in Florida, the fruit had slightly thicker peels (table 127). This was less noticeable in fruit grown on the heavier soils of coastal districts.

Factors That Affect Quality

QUALITY OF JUICE AND FRUIT was considered by Hodgson (1947) to be related to these factors: (1) climate—most important for both quality and size; (2) rootstock—next for quality; and (3) nutrition—next for size.

Benton (1941, 1944) expressed the opinion that improvement in the palatability of grapefruit would be largely dependent upon climate and rootstock. He said that in Australia, regions with fairly high temperatures during most of the year and with mild winters should be selected for the production of grapefruit of good to best quality.

Sites (1947) discussed the effect of weather on fruit quality. He stated that few growers realized how weather conditions could cause marked differences in quality—as great as or greater than those induced by any cultural or nutritional treatments used up to that time in Florida citriculture.

It thus appears that effects on fruit quality must be caused by combinations of weather conditions rather than by any single one.

The growth and maturation of citrus fruits are decisively influenced by the climate of the region in which they are produced. Temperature, moisture, and light are the 3 important factors that affect the growth of plants. These, together with wind velocity and atmospheric pressure, are collectively designated as "climate." All biochemical reactions occurring in citrus fruits are influenced by temperature, and temperature primarily determines the areas that favor commercial citrus production. For the various species of *Citrus,* the minimum temperature below which growth ceases or commercial production is impractical is approximately 55° F. (13° C.). The optimum temperature may sometimes produce excessive rates of growth, which condition is not beneficial for commercial production, but in most crops, some temperature near the optimum is required.

From a physiological viewpoint, temperature is the condition of a biological substance which determines the direction in which the flow of heat takes place. Thermal equilibrium is the state in which a body receives and gives off equal quantities of heat per unit of time. It therefore follows that radiation is the process of transferring heat from a source to other bodies.

Scora and Newman (1967) reported the monthly mean temperatures for the 6 major citrus-producing regions of the United States (figure 85). They used the Köppen-Geiger (1936) classification system, placing a label on each of the 6 temperature curves used to designate the climatic characteristics of the several areas. The curve for the Santa Paula (California) area is classed as "Csb," which indicates a condition similar to that in the coastal and intermediate valleys of southern California, with their humid, moderate, subtropical climate, cool, dry summers, and limited rainfall occurring in the late fall, winter, and early spring.

The Riverside area is classed as "Csa" which, as a result of warmer summers, represents the interior valleys of southern and central California, located between the coastal range and the high Sierra.

The citrus-producing areas of Florida have a humid, subtropical climate which is classed as "Caf" as a result of abundant precipitation throughout the year. Although Florida has a mean annual temperature of 21° C. (70° F.) or more in nearly all citrus-growing areas, Scora and Newman noted that the southern tip of the peninsula had approximately the climate of a tropical rain forest ("Am").

The climatic factors of the Lower Rio Grande Valley, as represented by Weslaco (Texas), were definitely classified as "BSh," indicating a warm, semiarid, subtropical, steppe climate. The "BSh" notation also requires a rainy season that supplies abundant soil moisture for vegetative growth during a particular season of the year, followed by an intensely dry season of sufficient duration to eliminate the active growth of all perennial deciduous vegetation. The mean annual temperature of this region is about 22.8° C. (73° F.). Under

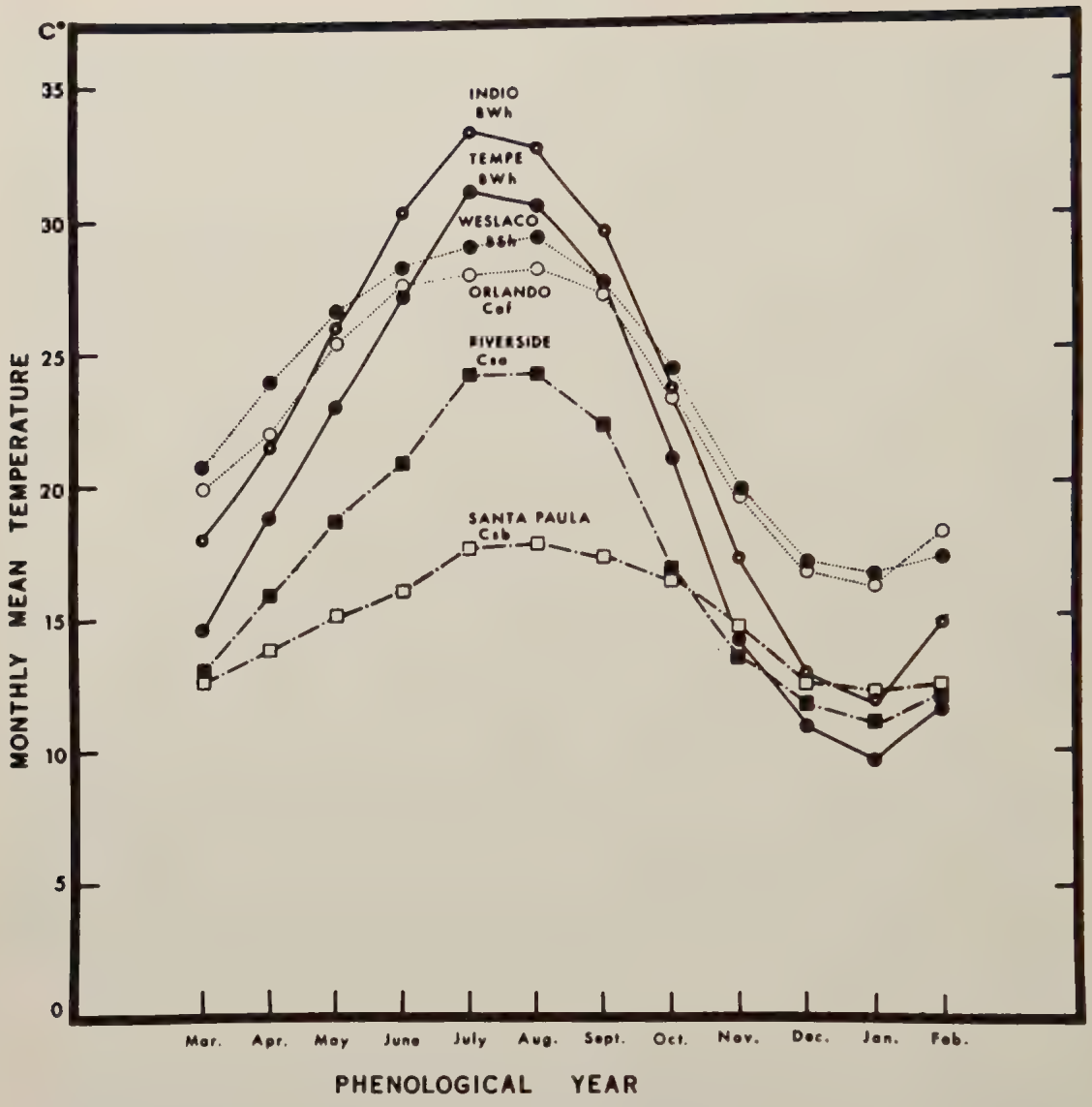


Fig. 85. Monthly mean temperatures for 6 locations in major citrus-producing areas in the United States. Derived from data furnished by the United States Weather Bureau. (From Scora and Newman, 1967.)

these climatic conditions, the annual grasses become the natural climax vegetation. The rainy season in the Rio Grande Valley is limited to late summer, fall, and early winter, with the maximum precipitation occurring in September and October.

According to the Köppen-Geiger classification system, the lower deserts of California and Arizona are designated as "BWh," indicating a warm, subtropical desert with a mean annual temperature not lower than 17.7° C. (64° F.). In contrast, if the mean annual temperatures were below that figure, the areas would be designated as cold deserts ("BWk"). The Coachella Valley of southern California has a mean annual temperature of 22.2° C. (72° F.), and the Salt River Valley near Phoenix, Arizona, has a mean annual temper-

ature of about 19° C. (66° F.), which is a little above the "cold desert" classification.

Although the curves illustrated in figure 86 were determined on Valencia oranges by Scora and Newman (1967), they are applicable to grapefruit in that they reveal the comparative rates of growth and maturation of citrus fruits, as indicated by the seasonal changes in the ratios of percentages of total soluble solids to titratable acidity expressed as anhydrous citric acid. The time-sequence curves of the ratios indicate significant differences in the rates of maturation of citrus fruits from the 6 major citrus-growing regions of the United States. The variations in rates are highly correlated with the mean monthly temperatures of the phenological year (figure 85). In the coastal area of California (Santa Paula), which has cool summer months, Valencia oranges required approximately 17 months after the mean blooming date in the preceding year to attain commercial maturity. At Riverside (Cal-

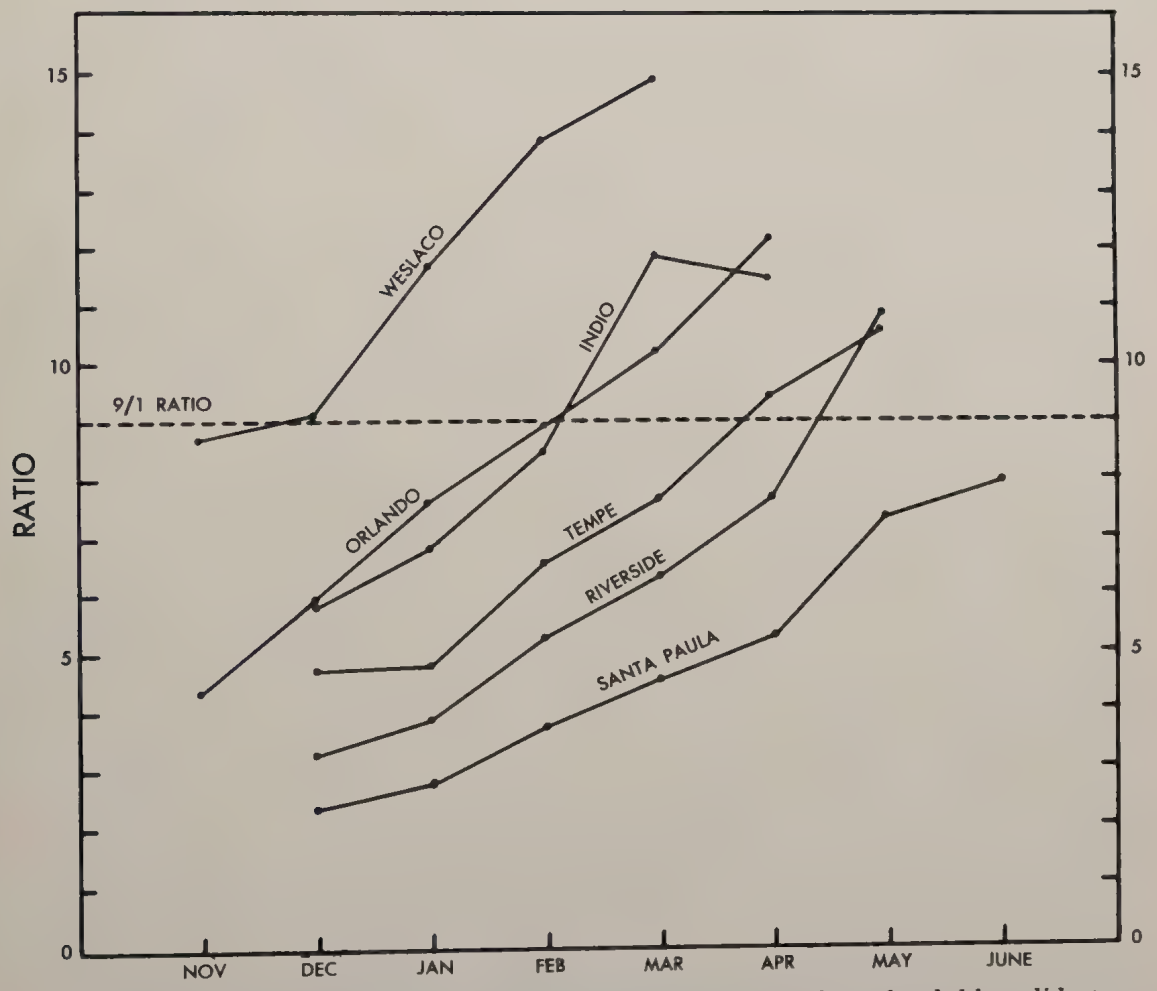


Fig. 86. Seasonal changes in the ratios of percentages of total soluble solids to percentages of total acids in Valencia orange juice in major citrus-producing areas in the United States. (From Scora and Newman, 1967.)

ifornia), the higher mean temperatures in this inland district during the late spring and summer produced favorable conditions for increased photosynthetic activity, and resulted in increased accumulation of soluble carbohydrates in the fruit, thus producing a maturity ratio of 9 to 1 in about 13 months after the mean blooming date. The maturity curve of Valencia oranges grown in Weslaco (Texas) reached a ratio of 9 to 1 in late November, and the curves for Orlando (Florida) and Indio (California) reached 9 to 1 in mid-February, approximately 11 months after bloom. Tempe (Arizona) has a slightly lower mean monthly temperature than Indio; consequently, the time required to mature Valencia oranges (9 to 1) in this desert region was 12 months (late March).

Sinclair and Bartholomew (1944) reported that grapefruit is outstanding in the differences of time required for fruit to mature under different climatic conditions. Buds on grapefruit trees come to full bloom and set fruit at approximately the same time at Riverside ("Csa" classification) and at Brawley (lower desert of southern California), but about 13 months are required to mature the fruit at Riverside, and only 7 to 8 months at Brawley. The shorter time required for grapefruit to reach maturity at Brawley is chiefly owing to the excessive heat units, which accelerate the reactions involved in growth and in the elaboration of food materials.

Within a limited climatic environment, the time interval between full bloom and maturity of citrus fruits would probably approximate a constant over a period of several years. In California, however, citrus is grown under widely different climatic conditions, ranging from the very hot, dry climate of the desert valleys to the temperate, rather humid climate of the coastal regions. As already noted, such differences in climatic conditions will markedly affect the rates of biochemical reactions occurring in the fruit during development and maturation. This means that the time interval between full bloom and maturity may be greatly altered by the climate. In connection with this problem, it should be borne in mind that the citrus tree is an evergreen, and does not have a rest period comparable to that of deciduous trees. Citrus has a very long growing season; some species, such as the lemon, may bloom and produce fruit the year around.

Sites and Reitz (1949; 1950*a*, *b*), in studying the effects of exposure on the rates of chemical changes in Valencia oranges, analyzed individually all the fruits from a tree, and correlated various chemical constituents with their positions on it. The total soluble-solids content was highest in the outside fruit (in an exposed location), intermediate in concentration in fruit located in the canopy of the tree, and lowest in fruit located on the inside. They also noted that the solids in the fruit increased with increased height on the tree. This characteristic was clearly associated with exposure to light. Turrell *et al.* (1964) confirmed these results by showing that fruit picked from the tops of

trees had lower moisture contents and higher peel-puncture pressures than fruit picked from their bottom areas.

When oranges from the same tree locations were graded for color, Sites and Reitz further reported that green fruit was associated with an inside location, low total soluble solids, and low vitamin C; yellow fruit grown under partially shaded conditions contained intermediate solids and vitamin C; and orange-colored fruit grown on the outside of the tree under exposed conditions had the highest total soluble solids and highest vitamin C. The exposure of leaves attached to the fruit-bearing stems is probably essential for the production of soluble carbohydrates by photosynthesis, which subsequently results in increased translocation of soluble constituents to the fruit.

The percentage of free acid in the fruit, sampled from various locations on the tree, did not vary as much as the total soluble solids. Consequently, it would be reasonable to conclude that the ratio of total soluble solids to acids would vary similarly to the total soluble solids in fruit obtained from different locations, and that the highest average ratios existed in the outside fruit, medium ratios in the canopy fruit, and the lowest ratios in the inside fruit.

Turrell *et al.* (1964), who recognized the importance of climatic factors in their relationship to the rates of growth and maturation of citrus fruits, collected grapefruit and orange samples from different citrus-growing areas in southern California, which included (1) coastal, (2) intermediate, (3) interior, and (4) desert climatic regions, for the purpose of obtaining morphological and chemical data relative to peel tenderness and other fruit characteristics. The 4 climatic regions were characterized by their mean monthly air temperatures for June, July, August, and September for the 7-year period from 1949 through 1955. The values were computed from the mean daily temperatures, which were the averages of the daily maxima and minima of 9 weather stations in the coastal area, 10 in the intermediate, 9 in the interior, and 6 in the desert. The 7-year, 4-month mean temperatures for the 4 regions amounted to (in rounded figures) 66, 71, 73, and 87° F., respectively (19, 22, 23, and 30° C.).

The differences in temperatures between districts were highly significant statistically. In this study, the mean air temperatures in the areas in which the fruit was grown were similarly computed for the 4-month period from the weather data of the station nearest to the sampling plots. These temperatures proved to be very close to the 7-year and 1955 means for the respective districts, and they were significantly correlated with the 1955 mean ($r = +0.986$).

Temperature, humidity, and wind velocity are the climatic factors which affect the moisture content of the fruit, and these 3 variables can be adequately expressed in terms of the evaporating capacity of the air. In figure 87, the solid line shows the summer mean air temperatures in the 4 climatic

regions in which the fruit samples were collected in 1955, and the broken line shows the correlation ($r = +0.806$) between evaporation and air temperatures at the United States weather stations for the same period and the same areas. Since the mean air temperatures of the 2 curves in the figure are highly correlated ($r = +0.753$), the air temperatures in the sampling areas may indicate with a fair degree of accuracy the relative evaporating potential of the air (Turrell *et al.*, 1964).

The tenderness of grapefruit peel can be related to its moisture content by plotting the peel puncture-pressure values (psi) against the peel dry weight (grams) per unit volume of fresh peel (cubic centimeters). The increase in dry weight or the reduction in moisture content of the mature peel samples resulted in an increase in the pounds of pressure necessary to puncture the peels (figure 88). The correlation coefficients between these 2 variables were highly significant; as indicated for various citrus fruits, $r = +0.921$ for grapefruit, $r = +0.398$ for Valencia oranges, and $r = +0.904$ for Washington Navels. Turrell *et al.* (1964) made the interesting observation that the puncture pressure of the peel was often significantly correlated with the pulp dry weight

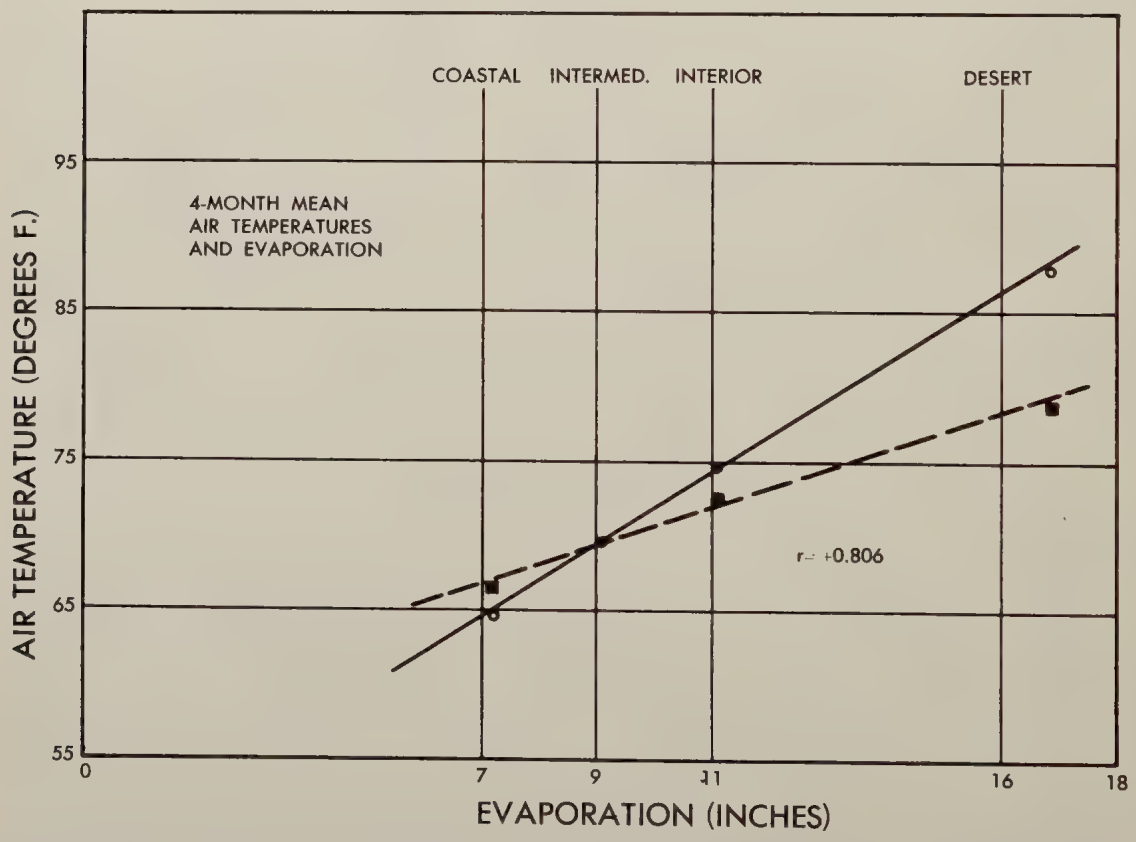


Fig. 87. Solid line indicates summer mean air temperatures in 4 climatic regions where fruit samples were collected in 1955. Broken line indicates the correlation of evaporation and air temperatures at United States weather stations for the same period and same regions. (From Turrell *et al.*, 1964.)

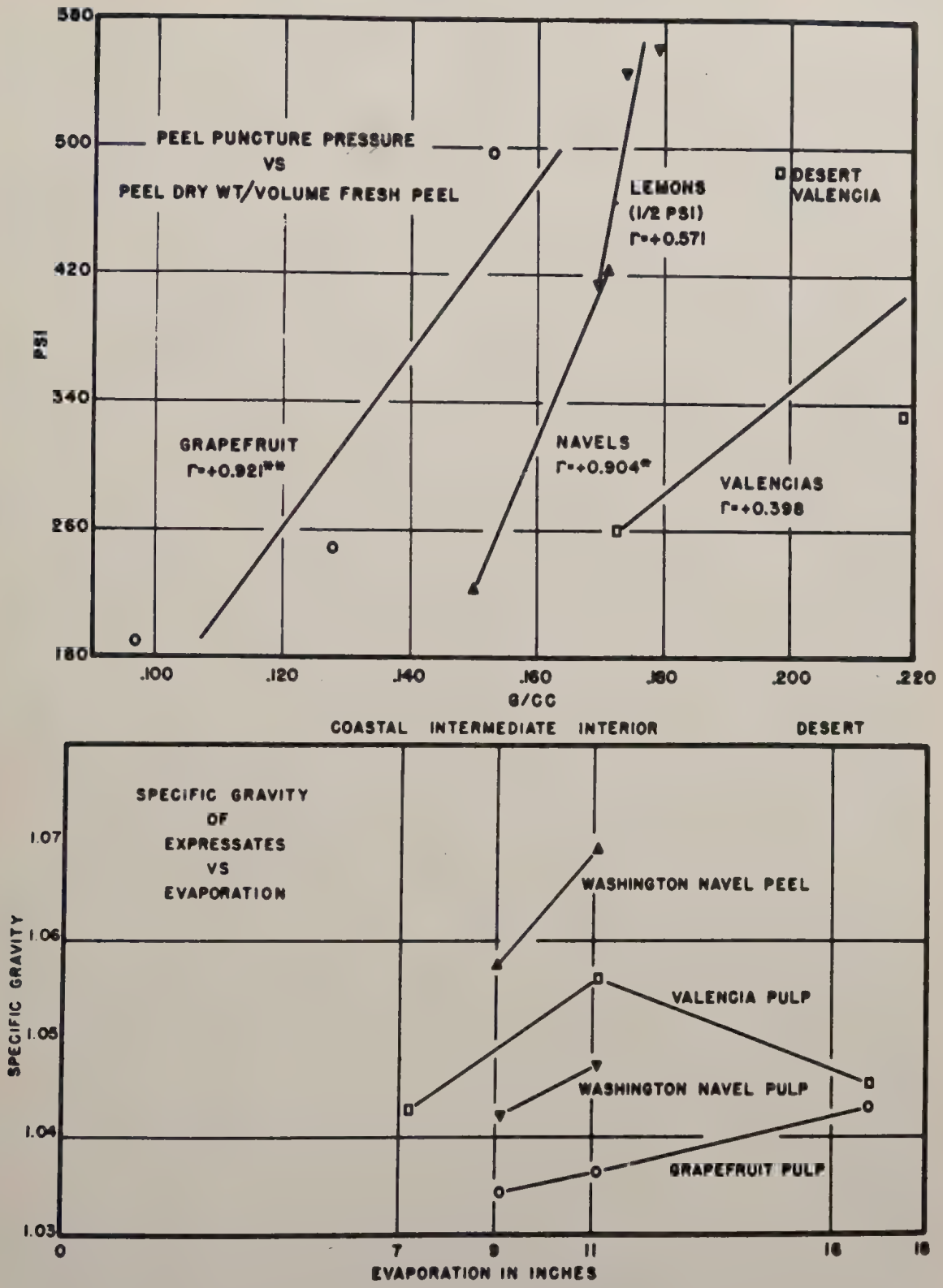


Fig. 88. *Top:* Correlation between peel puncture pressure and peel dry weight per unit volume of fresh peel. *Bottom:* Relationships of specific gravity of expressed liquids of peels and pulps and evaporation rates of 4 climatic citrus-growing areas in southern California. (From Turrell *et al.*, 1964.)

per unit volume of pulp per fresh weight—equivalent to the percentage of dry weight on a fresh-weight basis. With the exception of the lemon ($r = -0.339$), the degree of association for this relationship showed $r = +0.898$ for grapefruit, $r = +0.545$ for Valencia oranges, and $r = +0.920$ for Washington Navels. The puncture pressures of grapefruit peel were lowest in the intermediate climatic areas, moderate in the interior, and highest in the desert regions. The data obtained on all citrus varieties, except lemons, substantially supported the existence of a high negative correlation ($r = -0.768$) between peel puncture pressure and percentage of moisture (dry-weight basis). In the desert areas of California, Marsh grapefruit had an exceptionally high peel puncture pressure, and the latter values were positively and significantly correlated with the evaporation potential of the air ($r = +0.631$). The percentage of peel moisture (dry-weight basis) was negatively and highly significantly correlated with the evaporating power of the air ($r = -0.862$).

A close inspection of the lower graph in figure 88 will show the relationship between the specific gravity of expressed liquids of peels and pulps and the evaporation rates of 4 climatic citrus-growing regions in southern California. Peel expressates from the Washington Navel orange had lower specific gravities in fruits from intermediate areas than similar samples from the interior. The mean specific gravity of pulp expressates from Marsh grapefruit increased progressively from the moist intermediate regions to the dry desert areas. Similar data were obtained for both the Washington Navel and Valencia orange pulps, except for the Valencia variety in desert areas. In grapefruit, the dry weight of peel per unit volume of fresh peel was usually lower in fruit from the bottom than in fruit from the top of the tree. Concomitant with the peel dry-weight data, the specific gravity of pulp expressates from Washington Navel was significantly lower in bottom fruit than in top fruit from both intermediate and interior regions.

The high negative correlation between peel moisture and total evaporating power ($r = -0.862$) of the various citrus-producing areas can be amply demonstrated by comparing the curves constructed from the data of Sinclair and Bartholomew (1944) and Monselise and Turrell (1959), shown in figure 89 on the next page.

The close agreement between the data from these 2 sources strongly indicates that different climates do cause real differences in the moisture contents of citrus fruits.

Monselise (1959) noted that ripe citrus fruits, when grown and matured under widely different climatic or environmental conditions or on different rootstocks, exhibited a highly negative correlation between their peel moisture percentages and their resistance to peel puncture pressure. In his studies on the Shamouti (Jaffa) orange in Israel, Monselise detected no seasonal changes in the moisture percentages of the peel, but he did observe a contin-

uous increase in fruit volume with a simultaneous decrease in resistance of peel to pressure. This decrease was especially precipitous during the ripening period, but was less so after the fruit reached maturity. Peel thickness decreased very sharply in the preripening stage, but subsequently increased gradually. It was emphasized that the thickness of the peel was not generally correlated with its moisture content, but these 2 unrelated factors were together the cause of the lower resistance in the "tender" orchard peel. Such data seem to lead to the recognition that peel thickness is, in some instances, influenced by factors that are independent of moisture content.

Fruit peel from the "tender" orchards was thinner, and contained higher moisture content and lower resistance to pressure and bruises. Monselise (1959) found all these factors to be statistically significant to various degrees during the picking season of the Shamouti orange. Analyzing the data by the method of multiple covariance, he found that the total variance in resistance between 2 "tender" orchards could be attributed to the combined effects of peel moisture ($r = -0.538$) and peel thickness ($r = +0.446$), but no correlation between moisture and peel thickness was observed.

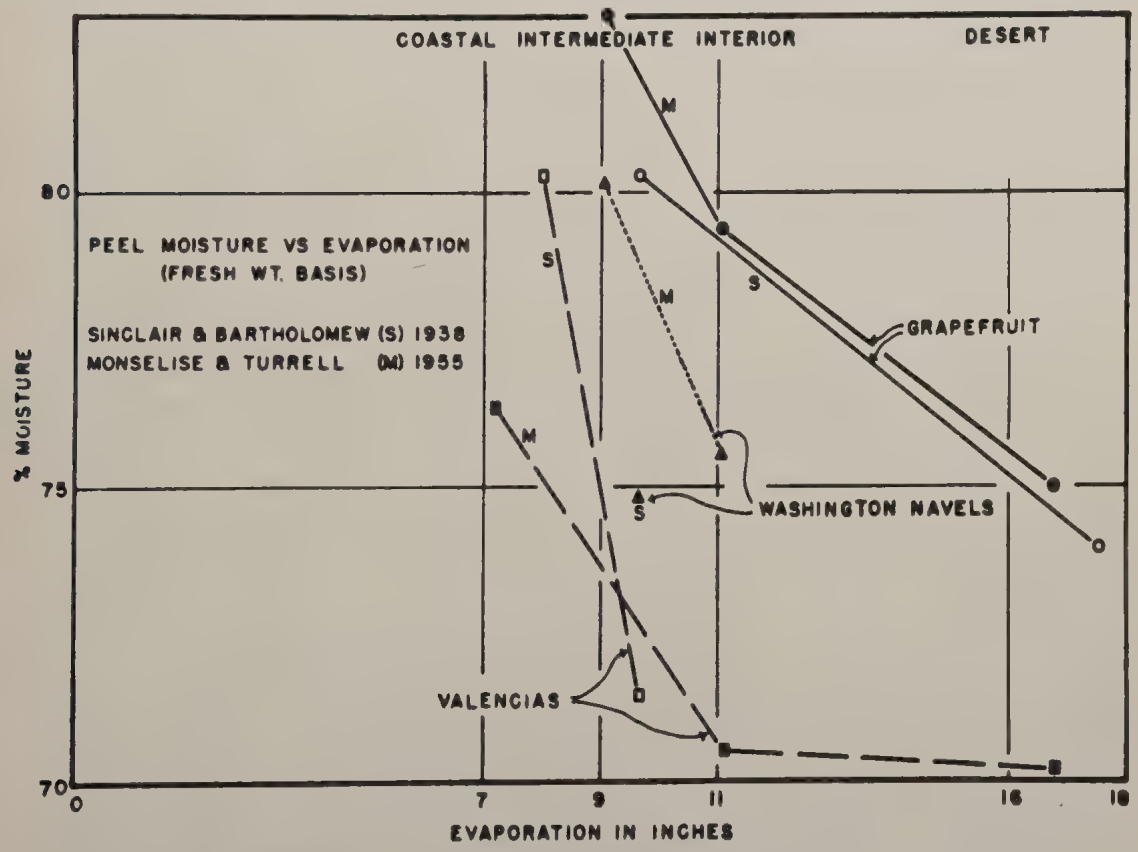


Fig. 89. Comparisons of peel-moisture contents (data from Sinclair and Bartholomew, 1944; Monselise and Turrell, 1959), and evaporation (data from the United States Weather Bureau in 1955) in 4 climatic citrus-growing regions in 1938 and 1955. (From Turrell *et al.*, 1964.)

With Valencia orange leaves used as assay material, Newman *et al.* (1967) measured the net heat accumulation, the degree–hour accumulation, and the time in days required to accumulate these heat characteristics that are so necessary for the growth and maturation of Valencia oranges grown in 5 commercial producing areas of the United States. These researchers emphasized the fact that the degree–hour accumulations (Part 1) were not so highly related to Valencia fruit growth and maturation as the net heat accumulations (Part 2) in table 128. The net heat accumulations were more highly correlated with the total time between full bloom and maturity than with the shorter periods of growth, as indicated by changes in fruit volume. It was also suggested that changes in the rate of fruit volume might not be good indices to the rate of fruit maturity. Inspection of the table (Parts 1 and 2) will show that both the net heat accumulations and the degree–hour accumulations were poorly associated with the various growth stages of the Valencia orange. This lack of association can be verified by measuring the degrees of variation between the experimental values. The mean standard error for the heat-unit accumulations, for 3 stages of growth for 4 crop years from 5 geographical locations, amounted to 2,634 calories, with a mean coefficient of variation of 21 per cent. Similar calculations for the degree–hour data showed coefficients of 40 to 60 per cent—an excessive amount of error.

From their fruit-growth data, which included the net heat accumulated from full bloom to maturity of the fruit for the total phenological crop years from all geographical locations, Newman *et al.* (1967) estimated a grand mean for the net heat accumulations of 72,730 calories per cm^2 per minute with a mean standard error of 1,911 calories, and a mean coefficient of variation of 5 per cent. With respect to the growth of Valencia oranges, the grand mean indicates that, on the average, a Valencia fruit should attain a maturity ratio of 9 to 1 upon the accumulation of 72,730 cal/cm^2 of net radiant energy above the base radiant heat load of 0.524 cal/cm^2 per minute.

Part 3 of table 128 gives the time in days required for Valencia oranges to reach each stage of development during seasonal growth, from bloom to maturity (ratio of 9 to 1).

In addition to temperature and moisture, light is a climatic factor that fundamentally affects the growth and development of plants. The 3 components of light are intensity, quality, and duration; all these are essential for photosynthesis, flowering, and seed formation. However, plants are highly variable in the amount of light required for full growth and development. Some plants require high light intensities, while others develop fully under low intensities. With respect to flowering behavior, plants are grouped into 3 photoperiodic classes: long-day, short-day, and day-neutral plants. Many species show specificity for light quality, producing more photoperiodic response with particular wave lengths.

TABLE 128

NET HEAT ACCUMULATIONS REQUIRED FOR THE PRODUCTION OF VALENCIA ORANGES FROM BLOOM TO MATURITY.*

(Crop season 1964–65.)

Part 1. The degree-hour accumulation between 55 and 95° F. (13 and 35° C.) for each major phenological growth period, and the total for each of 5 geographic locations of commercial Valencia orange production in the United States.

Locations	Bloom to 25 cm³ (hours)	25 cm³ to 100 cm³ (hours)	100 cm³ to 9:1 ratio (hours)	Bloom to 9:1 ratio (hours)
Orlando, Florida	23,284	51,259	45,966	120,509
Weslaco, Texas	33,022	63,162	46,756	142,940
Indio, California	22,259	75,431	25,060	120,750
Riverside, California	32,888	30,089	10,389	73,366
Santa Paula, California	20,515	18,377	21,366	60,258
Standard error	1,214	4,681	3,192	7,015
Coefficient of variation (per cent)	23	49	53	34

Part 2. The net heat accumulation in calories per square centimeter per minute above the mean radiant temperature of 50° F. (10° C.) for each major phenological growth period, and the total for each of 5 geographic locations of commercial Valencia orange production in the United States.

Locations	Bloom to 25 cm³ (cal)	25 cm³ to 100 cm³ (cal)	100 cm³ to 9:1 ratio (cal)	Bloom to 9:1 ratio (cal)
Orlando, Florida	14,065	26,270	32,397	72,732
Weslaco, Texas	17,245	28,036	23,835	69,116
Indio, California	17,838	37,457	22,395	77,690
Riverside, California	24,534	25,132	19,225	68,891
Santa Paula, California	23,270	19,686	26,611	69,567
Standard error	1,961	2,893	2,222	1,673
Coefficient of variation (per cent)	23	24	20	5

Part 3. Time in days for the degree-hour and net heat accumulations associated with phenological periods of growth and development, at 5 geographical locations of commercial Valencia orange production in the United States.

Locations	Beginning dates	Ending dates	Bloom to 25 cm³ (days)	25 cm³ to 100 cm³ (days)	100 cm³ to 9:1 ratio (days)	Bloom to 9:1 ratio (days)
Orlando, Florida	3/27/64	2/2/65	60	93	159	312
Weslaco, Texas	3/16/64	11/18/64	67	91	89	247
Indio, California	4/27/64	2/6/65	55	103	127	285
Riverside, California	5/18/64	4/13/65	91	116	123	330
Santa Paula, California	5/18/64	7/23/65	96	98	237	431

*From Newman *et al.* (1967).

Light intensity affects the growth of plants in relation to its role in photosynthesis. If plant life is to survive over a prolonged period, light intensity must be sufficient to fix carbon dioxide photosynthetically during the day equal in quantity to the amount used in the 24-hour respiratory period. The quantity of light intensity necessary for survival varies significantly among different plants, depending, of course, upon whether the species is a shade or sun plant. The different effects of temperature on the rates of photosynthesis at various light intensities can be noted by the fact that at low intensities, where light is rate-limiting, the photosynthetic rate is independent of temperature; but under conditions of high light intensity where carbon dioxide is rate-limiting, the rate of photosynthesis increases with increase in temperature. In applying these principles to the growth and development of citrus fruits, it is entirely possible that the blooming period in citrus, in widely different latitudes, may be partially influenced by the length of day, but most experimental evidence tends to indicate that seasonal temperatures are the dominant influences in citrus production.

In their studies on factors affecting the growth and development of citrus fruits, Bartholomew *et al.* (1950) reported that the concentrations of soluble solids and sugars and the ratios of soluble solids to acids of grapefruit were higher in the outside fruit (exposed) than in the inside fruit (shaded). The inside fruits, located in the interior of the tree where they were hidden by relatively dense foliage, always had lower concentrations of total soluble con-

TABLE 129

THE EFFECTS OF EXPOSURE ON THE WEIGHT, SIZE, AND NITROGEN CONTENT OF SHAMOUTI ORANGE FRUITS.*

Measured factors	Data at end of growing season		Mean differences, exposed–shaded (July to January)	Statistical significance (F values)†
	Exposed	Shaded		
Fresh weight (grams)	205.11	182.58	23.5	39.7**
Dry weight (grams)	25.04	22.72	2.51	41.6**
Volume (milliliters)	224.5	187.0	32.5	41.1**
Surface (square centimeters)	178.4	157.9	16.7	65.2**
Circumference (centimeters)	24.7	23.4	1.46	110.0**
Polar axis (millimeters)	79.0	74.1	4.85	98.3**
Equatorial axis (millimeters)	73.4	69.0	4.23	123.6**
Peel thickness (millimeters)	5.6	5.2	0.38	11.4**
Specific gravity	0.914	0.976	−0.021	15.1**
Total nitrogen (per cent of dry matter)	1.182	1.220	−0.041	4.32 NS
Protein nitrogen (per cent of dry matter)	0.687	0.673	−0.010	1.71 NS

*From Goren and Monselise (1964).
†Two asterisks equal "significant at the 1 per cent level"; NS equals "nonsignificant."

stituents and total sugars than did the outside fruits, which were fully exposed to sunlight on the surface of the tree.

Goren and Monselise (1964) made some interesting comparisons of the Shamouti orange grown under exposed and shaded conditions during the period from July to January. The data in table 129 represent mean values obtained from exposed and shaded oranges sampled during the experimental period. At the end of the growing season, the mean light intensity had a significant effect on the external and internal physical attributes of the fruit. Higher dimensions were determined in oranges exposed to solar radiation than in those grown in the shade. The authors pointed out that these differences might have been caused by greater photosynthetic activity of green peel at higher temperatures and higher light intensities.

Soil Types. Soil type has been found to affect the quality and production of citrus fruits. Between very coarse-textured soils at one extreme and clay or adobe soils at the other, there are many soil types that will produce grapefruit and other kinds of citrus of good quality. Soils of a very coarse texture often are to be avoided because of infertility caused by excessive leaching. On the other hand, clay or adobe soils may not contain sufficient oxygen to permit root growth and spread, and their slow drying may result in root decay. A soil having a uniform profile is to be desired, because in such a profile the movement of water and oxygen is not dangerously impeded.

As Camp *et al.* (1954) pointed out, the 2 general soil types on which citrus is grown in Florida are (1) the extremely light, sandy soils that contain a very small amount of organic matter and are acid in reaction, with slight buffer capacities; and (2) chiefly those heavier soils that are alkaline owing to the presence of excess calcium carbonate. The authors stated that, in sandy soils with pH between 5.25 and 6.0, many mineral deficiencies could be corrected by applications of magnesium, manganese, copper, boron, and frequently zinc. In Florida, a soil of pH 5.50 is generally recommended as sufficient to make the various mineral elements available to citrus trees, and thus reduce to a minimum the number of elements to be applied as sprays. In alkaline soils which contain large amounts of calcium carbonate, it is difficult to reduce the pH to between 5.50 and 6 for fear of solubilizing the calcium carbonate. Consequently, rather than change the pH of these soils, it is more desirable to adjust the fertilizer program to the pH.

In contrast to the soils of Florida's humid climate, the soils in the semi-arid areas of the southwestern United States have accumulated relatively high concentrations of soluble salts, so that some form of irrigation is needed to produce crops.

The water always evaporates from the plant leaves and soil surface, leaving behind the salt, which continually accumulates in the soil and is removed only by leaching with adequate amounts of irrigation water.

Jones and Embleton (1966) pointed out that semi-arid soils are not highly leached, and before cultivation and irrigation they contain most of the nutrients required for plant growth. In California, these soils are generally alkaline, and may or may not contain calcium carbonate; in some instances, they may contain excess salt. In comparing oranges grown under California (semi-arid) and Florida (humid) conditions, Jones and Embleton listed some of the comparisons and contrasts of Valencia oranges as influenced by both climate and fertilizers (tables 130 and 131). The climatic effects shown in table 130 were not altered by fertilizer treatments, but within each area, fertilizer practices produced a marked effect on fruit quality. The effects of nitrogen on fruit characteristics (table 131) were partially similar in the 2 areas, but the characteristics differed in degree and intensity. Under the semi-arid conditions of California, the decrease in yields (volume) did not occur from lack of nitrogen when the level in the leaves was maintained at or above 2.4 per cent (dry-weight basis). With an increase in leaf nitrogen above 2.4 per cent, the number of oranges per tree increased, with a corresponding decrease in fruit size. Although the experimental data were obtained on Valencia oranges, it is reasonable to assume that similar differences would occur in grapefruit if experiments were performed under the same climatic and fertilizer conditions as those used on the oranges.

The following text will help to show how soils affect fruit quality.

López-Domínguez and Cady (1921) and López-Domínguez (1919, 1921), in Puerto Rico, found that the percentage of juice was highest in fruit grown on clay soil. In 1921, however, López-Domínguez reported that this effect of soil type was not very noticeable late in the season.

Hardy and Rodríguez (1935), in Trinidad, found more juice in grapefruit growing on highly acid soils than on soils rich in lime. The juice of the lime-

TABLE 130

COMPARISONS AND CONTRASTS OF VALENCIA ORANGES GROWN IN CALIFORNIA (SEMI-ARID) AND FLORIDA (HUMID).*†

(Climatic effects.)

Factors	California	Florida
Season of harvest	February–October	February–June
Fruit size	Medium	Large
Fruit color	Deep orange	Light orange
Juice color	Deep orange	Pale orange
Rind thickness	Thick	Thin
Soluble solids	High	Medium
Citric acid	High	Medium
Juice (percentage)	Low	High

*From Jones and Embleton (1966).
†The climate is much less uniform in California than in Florida.

land fruit had a higher sugar:acid ratio, indicating better quality. Otherwise, there was little difference in the composition of grapefruit grown on the 2 types of soil. (See table 58, chapter 2.)

For more extended discussions of the types of soils best suited for the production of high-quality grapefruit and other citrus, see Reuther *et al.* (1968) and Chapman (1966).

Fertilizers. Citrus trees cannot keep producing commercial crops indefinitely unless 1 or more kinds of fertilizer are applied. In some areas where the soil is light, at least 1 kind of fertilizer must be applied when the trees are planted. The kinds and amounts to be used at any time can be determined by analysis of the fruit and leaf tissues and the appearance and amount of fruit produced. In many areas, nitrogen alone is necessary; in others, potassium and phosphorus must also be added. In still other areas, the so-called "minor elements," such as zinc, sulfur, copper, and boron, may be required. Some of these may be applied in organic form and others in inorganic form; some may be applied to the soil and others to the tree in the form of a spray. Chapman (1966) produced a comprehensive treatise dealing with the diagnostic data and criteria by which the fertility status of soils and the nutritional levels of plants can be appraised and controlled. The results of some published data as they apply to grapefruit are summarized in the following text.

Kinnison and Finch (1933) were among the earlier workers who studied the effects of special practices that might influence the nutritional balance on yield, texture, and time of maturity of grapefruit on the Yuma Mesa of Arizona. They applied 5 pounds of ammonium sulfate per tree after each irrigation, a total of 55 pounds per tree (about 2.25 and 25 kilograms). This

TABLE 131

COMPARISONS AND CONTRASTS OF VALENCIA ORANGES GROWN IN CALIFORNIA (SEMI-ARID) AND FLORIDA (HUMID).*
(Effects of increasing nitrogen.)

Factors	California	Florida
Fruit size	Reduced	Reduced
Fruit color:		
Color break	Slightly delayed	Slightly delayed
Regreening	Increased	Slightly increased
Orange intensity	Increased	Increased
Rind thickness	Marked increase	Slightly decreased
Soluble solids	Inconsistent	Increased
Citric acid	Inconsistent	Increased
Juice (percentage)	Reduced	Slightly increased
Ascorbic acid	Reduced	Inconsistent

*From Jones and Embleton (1966).

treatment caused larger fruit, higher acid and sugar content, and thus a low ratio. Fruit maturity was delayed.

Hilgeman *et al.* (1937) applied nitrogen in different forms to each of 6 plots, phosphate to 1 plot, and potash to another, also on the Yuma Mesa. They arrived at the conclusions shown here.

1. Seasonal differences in acids, thickness of peel, and yield per tree were far greater than differences caused by fertilizer treatments.

2. Thickness of peel seemed to be associated with the number of fruits per tree—the lower the yield, the thicker the peel, and the poorer the quality of the fruit.

3. The percentage of juice per fruit was not significantly affected by either fertilizer or seasonal conditions.

4. Consistent differences in fruit maturation were not caused by different fertilizers. but marked seasonal differences in quality were observed.

Camp and Fudge (1939), Camp (1943, 1945), Camp *et al.* (1954), and Fudge (1938, 1939, 1940, 1941) pretty generally agreed with Fudge and Fehmerling (1940) that in hammock groves in Florida, soils and rootstocks were of major importance in determining fruit quality and composition, but in groves on Norfolk sand, the heavy use of fertilizer, which had to be properly balanced with respect to both major and minor elements, exerted a preponderant influence. In their tests, the application of copper, manganese, zinc, and magnesium tended to improve fruit composition and quality. However, Fudge (1941) concluded that the comparatively large amounts of fertilizer that had to be applied to the soil to produce the relatively small changes in mineral composition of the fruit juice obtained were probably not warranted in practical grove culture.

In his study of the role of nitrogen in relation to yield and quality of grapefruit, Martin (1939), in Tucson (Arizona), obtained the results summarized in table 132. The trees in plots 1, 2, and 3, which had been given treatments to increase their nitrogen content before bloom, produced about 30 per cent more fruit than did those in plots 4, 5, and 6, which were not fertilized during the winter and had been further starved by a competing cover crop of barley. In a later paper, published in 1940, Martin made a similar report. He found that the best results were obtained when nitrogen was applied in winter, so that there would be a good supply in the tree just before the blooming period.

Many others have carried on tests to determine the effects of various inorganic and organic fertilizers on the quality and production of grapefruit and other types of citrus, only a few of which will be mentioned here.

Finch and McGeorge (1940, 1945), in Arizona, found that the results of their tests, as had those of others, gave evidence that nitrogen played an important role in regulating the fruitfulness of grapefruit trees. Potassium and phosphorus, either separately or in combination with each other or with nitrogen, seemed to have little effect on crop yield.

TABLE 132

EFFECTS OF FERTILIZER AND CULTURAL PRACTICES UPON THE YIELD AND QUALITY OF MARSH GRAPEFRUIT.*

Plot numbers	Winter treatment	Yields (per cent)†	Supplementary treatments		Percentages of each quality at harvest			
			Spring	Summer	No. 1 (Sunkist)	No. 2 (choice)	No. 3 (standard)	No. 4 (culls)
1.....	High N fertilization	196.8	High N	High N	13.7	27.8	34.5	24.0
2.....	High N fertilization	180.9	Starved	Starved	40.1	32.7	13.9	13.3
3.....	High N fertilization	192.8	High N	Starved	32.4	31.4	19.4	16.8
4.....	Starvation practices	148.8	Starved	Starved	39.9	34.2	11.5	14.4
5.....	Starvation practices	156.3	High N	High N	16.8	31.5	28.6	23.1
6.....	Starvation practices	138.8	Starved	High N	19.4	26.3	26.5	27.8

*From Martin (1939).
†Percentages of previous year's yield.

Cowart (1942) determined the effects of magnesium on the quality of Duncan and Marsh grapefruit. The experiment began in 1937, and determinations were made in January, 1941. All plots showed magnesium deficiency when tests began, and all were treated alike, except for the magnesium applications shown in table 133. The table shows that the application of magnesium caused increases in the substances of the 2 varieties; these were greater in Duncan than in Marsh. Cowart and Stearns (1941) obtained similar results.

Stearns and Sites (1943) stated that failure to apply adequate amounts of magnesium, zinc, manganese, and copper to the trees resulted in low soluble solids and acids, and in a delay in the fruit reaching the standard maturity requirement. The application of zinc, manganese, and copper, but not magnesium, resulted in an increase in soluble solids, but the acids showed little if any change.

Over a 5-season period, Sites (1944) applied nitrogen, potassium, and phosphorus fertilizers to 4 plots, and the same materials plus magnesium, zinc, manganese, and copper to 4 other plots of Marsh and Duncan grapefruit. The results for the Duncan plots are shown in table 134, which indicates that the plots receiving the supplemental elements showed substantially higher figures relating to citric acid, soluble solids, ratios, and ascorbic acid. They also had a sweeter taste and a general improvement in flavor and production. The increase in soluble solids was relatively greater than that of the acids, which resulted in higher ratios. The increases occurred in both varieties.

Jones (1944), in Arizona, stated that in order to have the proper amount of nitrogen in the trees at the proper time, enough mineral nitrogen was applied to them during the early winter (November and December) so that leaves of the spring growth would contain 2.2 per cent or more nitrogen (dry-weight basis). A grass cover crop was then grown, with the result that by late summer, the nitrogen in the leaves would be reduced to 1.3 per cent or less. He recommended perennial Sudan or giant panicum (*Panicum antidotale*) as good summer cover-crop grasses.

Jones *et al.* (1944*a*) pointed out that nitrogen no longer enters the fruit after it has reached maturity. It is the nitrogen that enters the fruit in excessive amounts during the preceding summer, while it is green and growing, that is responsible for its coarseness and other inferior qualities.

Finch (1944), also in Arizona, reported results similar to those of Jones (1944). He found that if the leaves had 2 per cent nitrogen at blossom time and continued to maintain 1.7 to 1.8 per cent, the fruit was likely to be rough and late in maturing, but if the nitrogen dropped to 1.4 or 1.3 per cent, the fruit would be of good quality. When the lower amounts were present, the yields of fancy and choice grades always increased. The fruits colored earlier and the degrees Brix and ascorbic acid were higher under low-nitrogen conditions while the fruit was growing and maturing.

TABLE 133

EFFECTS OF MAGNESIUM DEFICIENCY UPON THE COMPOSITION OF DUNCAN AND MARSH GRAPEFRUIT.*

Plots and treatments	Variety	Citric acid (per cent)	Total soluble solids (per cent)	Sugars (per cent)			Vitamin C (mg/100 ml juice)
				Reducing	Sucrose	Totals	
2, 11—No MgO.....	Duncan	1.68	10.55	3.19	1.96	5.25	39.6
	Marsh	1.42	9.02	3.14	1.86	5.10	35.0
3, 10—2 per cent MgO..	Duncan	1.69	11.04	3.23	2.44	5.80	42.7
	Marsh	1.50	9.62	3.40	2.25	5.77	36.8
4, 9—4 per cent MgO...	Duncan	1.76	11.58	3.80	2.82	6.71	45.6
	Marsh	1.52	10.00	3.56	2.23	5.91	37.1

*From Cowart (1942).

TABLE 134

MATURITY ANALYSES OF DUNCAN GRAPEFRUIT OVER A 5-SEASON PERIOD: FROM PLOTS FERTILIZED WITH NPK WITH AND WITHOUT MICRONUTRIENT SUPPLEMENTS, RECEIVING A 40 PER CENT ORGANIC MIXTURE PLUS SUPPLEMENTAL ELEMENTS; AND FROM THOSE RECEIVING A STRAIGHT NPK MIXTURE DEFICIENT IN SUPPLEMENTAL ELEMENTS.*

Seasons and treatments	Plot numbers	Juice weight (per cent)	Citric acid (per cent)	Brix	Ratios	Ascorbic acid (mg/100 ml)
1939–40						
NPK.....	6, 7	40.2	1.60	8.90	5.56	38.0
NPK + MgO + Zn + Mn + Cu	1, 12	43.2	1.74	10.55	6.10	49.3
1940–41						
NPK.....	6, 7	45.1	1.78	10.70	6.01	42.0
NPK + MgO + Zn + Mn + Cu	1, 12	42.8	1.82	12.29	6.75	47.8
1941–42						
NPK.....	6, 7	45.6	1.21	8.93	7.38	40.2
NPK + MgO + Zn + Mn + Cu	1, 12	49.2	1.32	9.83	7.45	41.5
1942–43						
NPK.....	6, 7	....	1.40	8.84	6.50	43.2
NPK + MgO + Zn + Mn + Cu	1, 12	....	1.49	10.93	7.37	51.2
1943–44						
NPK.....	7†	39.7	1.54	8.47	5.49	37.9
NPK + MgO + Zn + Mn + Cu	1, 12	38.9	1.68	9.68	5.77	42.3

*From Sites (1944).
†Plot 6 failed to bear sufficient fruit in 1943–44 to permit taking samples.

Jones *et al.* (1945) stated that the juice of fruit which matured under low-nitrogen conditions contained as much ascorbic acid at the end of the harvest season as did that from high-nitrogen plots at the beginning of harvest. They gave the following comparisons of the effects upon fruit quality of "continuing high nitrogen" in the soil as compared with "declining nitrogen."

(1) Higher nitrogen content of juice and peel. (2) Lower Brix and higher acid of juice (relatively lower Brix:acid ratio). (3) Higher pH of juice. (4) Lower ascorbic acid. (5) Later coloration of fruit. (6) Some regreening of fruit. (7) Larger fruit. (8) Heavier fruit. (9) Relatively low weight:volume ratio. (10) Relatively thick, coarse-textured peel.

In the genus *Citrus,* Jones and Embleton (1959) reported that the optimum nitrogen level for fruit quality might be deficient for yields. In much of the literature on this subject, deficient and excess utilizations of nitrogen have been defined largely in terms of yields, without noting other plant physiological characteristics. From a practical viewpoint, Jones (1966) defined deficient and excess nitrogen thus: "A plant is deficient in nitrogen so long as an increase in the nitrogen level will improve the economic returns of the plant, plant part, or plant product for which the crop is grown. A plant is excessive in nitrogen so long as a decrease in the nitrogen level will improve the economic returns of the plant, plant part, or plant product for which the crop is grown."

Innes (1946), in Jamaica, reported the following results of a 10-year test to determine the effects of fertilizers on the juice of Marsh Seedless grapefruit.

1. Nitrogen fertilizers on nitrogen-deficient soils decrease the ascorbic acid content, but increase the total soluble solids, acids, and sugars.

2. Superphosphate on phosphate-deficient soils decreases acids, sugars, and ascorbic acid, but increases the sugars:acid ratio.

3. Potash applied to potassium-deficient soils causes an increase in total soluble solids, pH, acids, total sugars, and ascorbic acid.

4. Potash plus superphosphate causes an increase in both yield and weight per fruit. Core and peel thickness are increased by potash, but both are reduced by superphosphate if the soil is deficient in that material.

Sites (1947), in discussing the results of his own tests and those of others to determine the effects of different fertilizers on fruit quality, stated that it had been conclusively demonstrated that improvement in quality would result from the correction of magnesium, manganese, zinc, and copper deficiencies. He emphasized that no benefit would be derived from the application of these elements in amounts exceeding those needed for the maintenance of internal fruit quality. He stated further that the use of these elements, especially on grapefruit, would not tend either to delay or hasten the time at which the fruit met state maturity requirements. These conclusions were in agreement with his own earlier ones (1944) and those of Cowart (1942).

Sites (1947) also determined the effects of potassium applications, without and with magnesium. The average percentages of soluble solids, in tests made at biweekly intervals, September 17–November 12, are shown here.

	Without magnesium	*With magnesium*
With no K_2O	8.77	8.42
With 3 per cent K_2O	8.88	8.79
With 5 per cent K_2O	8.33	8.68
With 10 per cent K_2O	8.47	8.96

Where magnesium was supplied, there was a fairly sharp rise in solids from the 0 per cent to the 3 per cent K_2O treatment, and a gradual increase from the 3 per cent to the 10 per cent treatment. Where magnesium was deficient, the solids fell off noticeably in the 5 to 10 per cent treatments.

Rose *et al.* (1951) cited some 15 or 20 articles on the effects of minor elements, such as zinc, magnesium, manganese, and copper; and then they stated: "The determination of the effects of minor and major nutrient elements on fruit composition under the widely varying conditions of rootstock, soil type, climate, and other environmental factors is difficult, and much critical research will be needed before final conclusions can be drawn." They noted that Cowart (1942), Cowart and Stearns (1941), Fudge and Fehmerling (1940), Skinner *et al.* (1934), Sites (1944, 1947), Stearns and Sites (1943), and Camp (1943, 1945), all working in Florida, had reported data which they interpreted as showing that improvement in fruit quality would result from the correction of manganese, magnesium, zinc, and copper deficiencies in soils on which citrus trees were growing.

Since a substantial portion of Florida's total citrus production is comprised of grapefruit, it follows that the effects of fertilizers on yield and fruit quality are of considerable importance to both producers and consumers. In order to maintain high standards of both yield and quality, researchers of the Florida citrus industry have published the results of many experiments on the responses of grapefruit to fertilizer treatments. In that area, grapefruit is usually planted on extremely sandy soils which are low in exchange capacity, and consequently retain only small quantities of exchangeable plant nutrients against the leaching action of irrigation and rain water. Since plant nutrients are held in the soil in varying degrees of availability, citrus soils must be treated with various amounts of nutritive elements in order to obtain high yields and good fruit quality.

In studying the effects of nitrogen on grapefruit grown on Lakeland fine sand, Sites *et al.* (1961) reported that both quality and yield of seedy grapefruit were found to be definitely affected by the rate and time of nitrogen application. They reported a highly significant positive correlation between the rates of nitrogen application and the percentages of soluble solids in the

juice, but they also reported a negative correlation between the rate of nitrogen applied and the yield of fruit per tree. It should be noted that the excessive nitrogen used in these experiments proved harmful, because there resulted not only a reduction in yield but also a reduction in tree vigor. Since the increments of nitrogen increase used in the rate treatments were excessive, the data should not lead to the conclusion that any nitrogen application is detrimental to yield. In these experiments, the optimum production, based on actual yield, occurred at 230 pounds (about 104 kilograms) of nitrogen per acre; consequently, applications above this amount resulted in decreased yields and reduced tree vigor. No attempt was made to explain the effects of an increased rate of nitrogen application on the increase of soluble solids and acid contents of the juice. Such pronounced differences in juice composition have not been observed in earlier data.

In the Indian River citrus area of Florida, Reitz and Hunziker (1961) carried out a 6-crop study of 5 rates of nitrogen application and their interaction with arsenic sprays on the quality and yield of Marsh grapefruit. The maximum production (9.66 boxes per tree) was obtained from application of 2 pounds (0.908 kilogram) of nitrogen per tree per year, and this particular amount of fertilization was equal to 0.21 pound (0.095 kilogram) of nitrogen per actual box produced (table 135). The average production in plots in

TABLE 135

THE EFFECTS OF NITROGEN RATE ON THE YIELD AND FRUIT QUALITY OF MARSH GRAPEFRUIT OVER A 6-CROP PERIOD, 1955–61.*

Factors	Pounds nitrogen per tree per year†					Statistical significance‡
	0	1	2	3	4	
Yield (boxes per tree)	4.94	7.02	9.66	8.99	8.43	**
Juice quality:						
Soluble solids (degrees Brix)	9.67	9.63	9.19	9.44	9.69	**
Acid (per cent)	1.35	1.31	1.32	1.36	1.44	**
Brix:acid ratios	7.23	7.41	7.04	7.06	6.98	NS
Juice content (per cent)	44.7	48.2	49.0	48.9	48.6	**
External quality:						
Fruit size (grams per fruit)§	516	533	535	492	478	*
Green fruit (per cent)‖	36.9	46.2	74.9	76.9	79.9	**
Coarse fruit (per cent)¶	26.7	26.8	26.6	35.4	31.8	NS
Per cent, longitudinal, of transverse diameter‖	91.5	92.3	91.5	92.6	93.0	NS

*From Reitz and Hunziker (1961).
†One pound equals 0.454 kilogram.
‡Two asterisks equal "significant at the 1 per cent level"; a single asterisk equals "significant at the 5 per cent level"; NS equals "nonsignificant at the 5 per cent level."
§Five-season averages. No data were collected for 1957–58.
‖Averages of the 1955–56 and 1956–57 crops.
¶Averages of December 1955 and 1956, and February 1959.

which no nitrogen was applied amounted to 4.94 boxes per tree per year. Nitrogen application in excess of 2 pounds per tree per year resulted in a decrease in fruit yield. In relation to internal fruit quality, the fruit from plots receiving 2 pounds of nitrogen per tree per year had the lowest levels of soluble solids in the juice. In other words, higher soluble solids were obtained at both lower and higher rates of nitrogen application. Juice acidity was increased by all except the first increment of application. Although the no-nitrogen treatment fruit (controls) was notably low in juice content, the plots receiving the various nitrogen treatments produced fruit with no significant differences in juice content.

On the basis of external fruit quality, the table indicates that there was a slight tendency for the fruit size to decrease with an increasing rate of nitrogen applied. However, the percentage of green fruit increased with a corresponding increase in the rate of nitrogen application. There was some indication that the degree of coarseness in the fruit, such as wrinkling or development of a shoulder about the stem end, increased as a result of high nitrogen use.

These investigators reported that after 4 months of the differential treatments, visible differences were observed in the trees' appearance. Trees receiving no nitrogen were both lighter green and had thinner foliage than those that received the treatments. After 1955, the foliage of the nontreated trees was extremely thin and yellow. The trees receiving only 1 pound (0.454 kilogram) of nitrogen per tree per year also had thin, yellow foliage. Those receiving 2 pounds per year maintained high-density, dull-green foliage. Throughout the experimental period, the trees receiving either 3 or 4 pounds annually showed no significant differences in the density or dark-green color of the foliage. Those plots which received no nitrogen and 1 pound of nitrogen per tree had some trees which showed not only severe nitrogen deficiency, but also symptoms of molybdenum deficiency.

During the 3 years of arsenic sprays, no statistically significant interaction between nitrogen rate and arsenic effect was observed on any fruit characteristic. In these experiments, arsenic was found to be equally effective at all nitrogen levels. As would be expected, the most important change as a result of the use of arsenic sprays was a decrease in the acidity of the juice, which demonstrated its practical significance in increasing the ratio of soluble solids to acid.

In an 8-year field experiment, Smith and Rasmussen (1961*a*) studied the effects of nitrogen sources, rate of application, and influence of pH on the production and quality of Marsh grapefruit. The application of nitrogen at rates of 120, 240, and 540 pounds per acre (54.5, 109, and 245 kilograms per 0.405 hectare) produced an average yield of slightly more than 500 boxes per acre, irrespective of the amounts used in the experiments. No significant

differences could be observed in the yields, fruit quality, or growth of the trees when their nitrogen requirement was supplied with 1 of the following 3 sources: calcium nitrate, ammonium nitrate, or ammonium sulfate. High nitrogen levels reduced fruit weight and increased soluble solids and total acidity, with an insignificant effect on the maturity ratios. The differences just noted were further accentuated at extremely high nitrogen treatments. In 5 of the 8 seasons, the vitamin C content of the juice was slightly reduced at higher nitrogen levels.

In these experiments, the only favorable characteristic of high-nitrogen treatments was the increase in soluble solids of the juice. On the contrary, the undesirable attributes were decreases in fruit size, increases in acidity of the juice, and delays in color break of the fruit. In general effect, excessive nitrogen had a tendency to lower grapefruit quality.

Smith and Rasmussen also reported results showing that arsenic toxicity in leaves was clearly associated with the nitrogen level. As a result of arsenic sprays containing 1.25 pound of lead arsenate per 100 gallons (0.567 kilogram per 379 liters), the leaves from the low-nitrogen trees showed extreme mottling every year. Trees in plots with intermediate nitrogen levels had leaves with an intermediate degree of arsenic injury. On the other extreme, the high-nitrogen trees usually showed no symptoms of arsenic injury to their leaves but, in some years, exceptionally mild symptoms did appear.

Smith (1963) determined the quality of selected sizes of grapefruit from trees differentially fertilized with nitrogen and potassium during an 8-year experimental period. The 5 sizes of grapefruit used in these experiments ranged from 176 to 70 per box. It was noted that both fertilizer treatment and fruit size showed some consistent effects on various fruit-quality factors. The lack of interaction between fruit size and fertilizer treatment indicated that adequate sampling of either selected or random-sized fruit would reveal the effect of fertilization on grapefruit quality. The nitrogen and potassium effects on fruit quality, determined on samples chosen from either selected or random-sized fruit, gave consistent results over the 8-year period. With an increase in fruit size, a decrease occurred in the percentage of juice, total soluble solids, ratio of solids to acids, and vitamin C concentration. In contrast, peel thickness and juice acidity increased.

Smith (1966) presented data which showed the effects of different rates of ammonium nitrate on the leaf and fruit characteristics of Marsh grapefruit on sweet-orange rootstock (table 136). The leaf nitrogen increased with the increased rate of nitrogen application, which subsequently resulted in a corresponding increase in fruit yield in boxes per acre. The author suggested that the nitrogen level below which yield would begin to decrease significantly appeared to be about 2.4 per cent (dry-weight basis) in leaves 4 to 7 months old from nonfruiting shoots. In these experiments, the rate of nitrogen ap-

plication had little effect on fruit characteristics considered detrimental to fruit quality, with the exception of vitamin C, which showed a slight decrease. The increased rate of nitrogen application reduced the rate of loss of green color in the peel with the approach of maturity, but the nitrogen levels that were used caused only a few days' delay in color development.

In experiments on the effects of timing of nitrogen applications on Florida grapefruit, Smith (1966) also reported data which showed the annual fruit production in boxes per acre, as influenced by varying the amount of ammonium nitrate used with the time of year in which it was applied (table 137). These data seem to indicate that extreme variation at the time of nitrogen application had little effect on either the quantity or quality of grapefruit produced. In other words, trees that received their total annual allotment of nitrogen at either the prebloom or postbloom stage (or at any other time) performed equally well with respect to yield. This was the case even at the lowest rate of nitrogen (50 pounds per acre), which finally resulted in partially defoliated, yellow trees.

The visual symptoms of potassium deficiency of grapefruit trees usually consist in dieback of the topmost leaves, irregular leaf changes from pale to bronze color, and the production of characteristically small fruit. Smith (1966) noted that deficiency, in terms of adverse effects on yield or tree condition, occurred at a potassium concentration of up to 0.7 per cent.

TABLE 136

EFFECTS OF DIFFERENT RATES OF AMMONIUM NITRATE ON LEAF NITROGEN, YIELD, AND VARIOUS FRUIT-QUALITY FACTORS OF MARSH GRAPEFRUIT ON SWEET-ORANGE ROOTSTOCK, 1960–65.*

Factors†	Nitrogen per acre (pounds)‡				
	50	70	90	120	Statistical significance§
Leaf nitrogen (per cent)	2.26	2.33	2.39	2.43	5
Yield (boxes per acre)	353	383	421	439	5
Fruit diameter (centimeters)	9.9	9.9	9.8	9.7	0
Rind thickness (millimeters)	6.3	6.3	6.3	6.3	0
Juice (per cent weight/weight)	54	54	54	55	0
Soluble solids (per cent)	8.90	8.90	8.90	8.90	0
Acids (per cent)	1.31	1.31	1.32	1.33	1
Ratios: soluble solids:acid	6.8	6.8	6.7	6.7	0
Vitamin C (milligrams per 100 milliliters)	39	39	38	37	3
Pounds nitrogen (per box)	0.16	0.18	0.20	0.26	..

*From Smith (1966). Also, unpublished data of author from experiment in progress.
†Stated as means for 5 years.
‡One pound equals 0.454 kilogram; one acre equals about 0.405 hectare
§Number of years that trend was significant at odds of 19:1.

In Marsh grapefruit grown under Florida conditions, Smith and Rasmussen (1961*b*) noted that chlorotic leaves showed a potassium content of 0.2 to 0.25 per cent. The deficiency was characterized by chlorosis that started as a blotched area from which the chlorophyll faded and which later turned from yellow to bronze. The distal half of the leaf was affected more than the basal portion. The symptoms developed in late summer and fall on leaves at least 5 to 6 months old, and persisted for several months with only slight leaf drop. The chlorotic leaves were also deficient in nitrogen and phosphorus, and the suggestion was made that this condition was the result of potassium deficiency.

For Florida citrus, a desirable range of potassium concentration has been said to be from 1.2 to 1.7 per cent (dry-weight basis) in the leaf. However, in past years the commercial groves of Florida have had a leaf potassium content higher than 2 per cent. Smith (1966) reported that under a humid climatic environment, potassium concentration had more effect on fruit quality than any other nutritional element, but that a wide range in leaf potassium could exist with insignificant effects on the quantity of fruit produced. When excessive rates of potassium were used, 20 to 30 per cent of the crop sometimes had to be eliminated from the fresh-fruit markets because of excessively large-sized and coarse-textured fruit. On applying a substantially reduced amount of potassium to the same groves, the fruit produced showed less than 1 per cent oversize, based on the same grading system. Consequently, 20 to 40 per cent of the fruits might have been undesirably small for the fresh-fruit market, but were highly satisfactory for conversion to citrus concentrate because they contained higher juice yields and were better in quality than the larger sizes; i.e., the smaller size produced higher yields of solids per acre.

TABLE 137

MEAN BOXES OF FRUIT PRODUCED PER ACRE ANNUALLY AT VARIOUS RATES OF AMMONIUM NITRATE AND DIFFERENT TIMES OF APPLICATION FOR 5 YEARS, 1961–65.*

When applied	Nitrogen per acre (pounds)†				
	50	70	90	120	Time means‡
All in January	346	386	449	419	400
All in April	347	365	383	451	387
All in July	344	378	395	441	390
All in October	347	424	438	431	410
Half in October; half in April	382	360	441	452	409
Rate means	353	383	421	439	...

*From Smith (1966). Also, unpublished data of author from experiment in progress. (One box equals 85 pounds of grapefruit—about 39 kilograms.)

†One pound equals 0.454 kilogram; one acre equals about 0.405 hectare.

‡Timing means do not differ significantly, by odds of 19:1. Extremes in rate means are significant.

The data in table 138 show the relationships between the concentration of leaf potassium and various fruit-quality characteristics of grapefruit produced under humid climatic conditions. The low values of leaf potassium were not associated with reduced yields. The data revealed specifically the effects of fruit quality as influenced by a wide range of leaf potassium that was induced by different fertilizer treatments used by various researchers. It can be observed that potassium produced larger fruit, increased rind thickness, and caused a higher concentration of acids in the juice. The yield of juice was decreased, with a possible reduction in soluble solids.

The reductions of both juice content and soluble solids are important attributes in fruit that is converted to juice products. As Smith (1966) noted, excessive applications of potassium to the soil can decrease the yield of juice products that are standardized on the basis of soluble solids.

Under the semi-arid conditions of California, phosphorus is deficient in very limited areas (Jones and Embleton, 1966), but under the humid climatic conditions of Florida, phosphorus deficiency has never been a serious problem in citrus production (Smith, 1966).

The soils used by orchardists in Florida usually have sufficient phosphorus for growth and fruit production of citrus trees. As Smith pointed out, relatively large amounts of phosphorus had accumulated in Florida citrus soils as a result of the customary use of phosphorus-rich fertilizers long before adequate recommendations were developed. Consequently, no phosphorus should be applied unless soil and leaf analyses show a deficiency. With respect to fruit quality, the fertilization of citrus trees with an excess amount of phosphorus tends to affect fruit composition adversely by lowering the concentrations of soluble solids and the vitamin C content. Phosphorus slows the rate of disappearance of green color early in the season of maturity, and hastens the regreening of fruit in late varieties that may remain attached to the tree for some time beyond the first year.

TABLE 138

ASSOCIATION OF POTASSIUM STATUS WITH VARIOUS FRUIT-QUALITY ATTRIBUTES OF GRAPEFRUIT GROWN UNDER HUMID CLIMATIC CONDITIONS.*

Leaf K range (per cent)	Fruit size	Green color	Rind thickness	Juice (per cent)	Acids (per cent)	Solids (per cent)
0.50–2.00†	++	(?)	+	(?)	++	+
.78–2.35	+	(?)	+	−−	++	+
.71–2.2	++	0	++	−	++	0
.70–2.20	++	0	++	−	++	0

*Adapted from Smith (1966). Other citations for data, in the vertical order given, are: Innes (1946); Deszyck and Koo (1957); Smith and Rasmussen (1960); and Smith (1963).

†Key to symbols: Plus signs, positive relationships; minus signs, negative relationships; zeros, no relationships; and (?), no data given. Each plus or minus, single or double, reflects a difference of about 5 per cent.

In a 20-year experiment on Pineapple oranges planted in virgin sandy soil, both leaf and soil analyses indicated that without added phosphorus, there was only a slight possibility of developing a critical deficiency of this element for many future years (Smith *et al.,* 1963). During the same study, with different rates of phosphorus varying between 18 and 144 pounds per acre (8 and 65 kilograms per 0.405 hectare) per year, most of the fruit responses were of small magnitude which had slight commercial importance. The reduction in acidity of the juice was the only compositional factor that could be considered beneficial, as this slightly advanced the date of maturity by increasing the ratio of soluble solids to acids.

Visible nutritional symptoms of the micronutrient or trace elements are not so easy to anticipate or determine as are those of the macronutrient elements. The chief difficulty of this problem revolves around the uncertainty of the plant absorbing micronutrients that have been applied to the soil. The micronutrient elements do not directly affect the yield or quality of citrus fruits. As Smith (1966) noted, deficiencies of these elements have been recognized in commercial citrus orchards of Florida, but the difference between a deficient and a sufficient amount of a trace element is exceedingly small. Consequently, experimental procedures are not sufficiently refined to measure a graduated response during the recovery of trace-element deficiencies in citrus trees.

It should be noted that the heavy-metal groups of micronutrient salts show some strikingly antagonistic effects. Reuther and Smith (1954) showed that toxic accumulations of copper in the soil interfered with the uptake of iron by citrus trees, and could induce severe iron deficiency symptoms. Other data have indicated that very high levels of copper, zinc, and manganese applied to liquid, sand, or vermiculite cultures produced some strikingly antagonistic effects between ion pairs on concentration in leaves, but that copper accumulated in the roots to the level of toxicity without being transported to the upper parts of the plant in increased amounts (Smith and Specht, 1953; Smith, 1956).

Irrigation. Very little research work appears to have been done to determine the relative growth and chemical composition of grapefruit from irrigated and nonirrigated groves. Such tests, of course, can be made only in areas where the trees and fruit can grow reasonably well without irrigation water to supplement the rainfall. Florida has such areas, and it was in these areas that most of the following tests were made.

Sites (1950*a, b*), after a 2-year test, reported that fruit quality was not found to be improved by the use of supplemental irrigation. The increased moisture caused by irrigation produced a decrease in total soluble solids and acids, and possibly in ascorbic acid, but an increase in granulation ("dry-end"), as shown on the next page.

1. On irrigated plots, 61.8 per cent of all fruits were dry to one-half inch (1.3 centimeter) from the stem end.

2. On nonirrigated plots, 35.3 per cent of all fruits were dry to one-half inch from the stem end.

3. On irrigated plots, 10 per cent of the fruits were without 1 or more dry segments.

4. On nonirrigated plots, 27.9 per cent of the fruits were without 1 or more dry segments.

About 2 years later, Sites *et al.* (1952) reported the following conditions as a result of irrigation and nonirrigation, using Silver Cluster and Marsh grapefruit. Tests were made on September 17, October 29, and December 2, 1948. The average over-all percentages of soluble solids are given here.

Silver Cluster		Marsh	
Not irrigated	9.50	Not irrigated	9.47
Irrigated	8.49	Irrigated	8.47

Sites and his associates drew the following conclusions, based upon the foregoing data.

1. Trees grown under conditions of continuous, abundant soil moisture produced fruits high in juice and low in soluble solids and acids. The reduction in soluble solids and acids was largely caused by dilution of the juice.

2. Soils with low moisture content produced juice moderately high in soluble solids and high in acids. This condition prevailed in the middle part of the season, when the soil moisture content was relatively low.

3. Low soil moisture content in the later part of the season had little or no effect.

4. Continuous irrigation increased granulation and dryness in the pulp at the stem end of the fruit.

5. A dry period of 3 months at any time after the fruit was set caused a reduction in its size. This reduction was never entirely regained by subsequent irrigations.

Savage (1951) approached the question from the economic rather than from the quality point of view. He pointed out that during years of low fruit prices, nonirrigated groves showed a greater net return than irrigated ones. For example, the average returns above operating costs for the period from 1941 through 1950 were $14.00 more per acre for nonirrigated groves than for irrigated ones.

Both Sites and Savage enumerated several factors, such as operating costs, type of fertilizer, and time of water application, which determine whether irrigation water should be applied. There are groves, perhaps in substantial numbers, in which irrigation has been profitable.

Ziegler (1955) reported the results of experiments conducted on Marsh grapefruit planted on Lakeland fine sand in central Florida. He found that irrigation treatments increased the fruit yield (boxes per tree), but that the increased yields were of statistical significance only in years of light rainfall. The data indicated that the total soluble solids per box and the percentage of total soluble solids in the juice were reduced by irrigation treatments. The

use of irrigation as a regular commercial operation, carried out under the conditions of these experiments, would be of doubtful economic value except in those years of low spring rainfall: less than 20 inches (50 cm) of precipitation during the January–June period.

Koo (1963) carried out a 3-year (1960 through 1962) experiment on the effects of frequency of irrigation treatments on yields of oranges and grapefruit. In the present review, particular attention will be paid only to his grapefruit data. The varietal response to irrigation was demonstrated by the fact that the Marsh grapefruit trees produced the highest yields to supplemental irrigation in terms of actual boxes produced and the percentage of yield increase, while the Pineapple orange gave the least response, with Valencia and Hamlin oranges showing intermediate results.

The effects of supplemental irrigation on fruit production were found to be related to the annual rainfall. This relationship was borne out by the fact that the largest increase in fruit yield from the irrigated treatments occurred in 1961, when the annual precipitation was 18 inches (45 cm) and less below normal, while the smallest increase took place in 1960, when the rainfall was more than 18 inches above the annual mean. Koo's data indicated that fruit yields were substantially increased by irrigation. The soil used in his experiment had a very low water-holding capacity. It follows that various soils retain different amounts of water at field capacity and wilting point; i.e., they have different water capacities. It was maintained that lower soluble-solids and acid contents of the fruit resulting from irrigation treatments applied later than early September appeared to be largely dilution effects. Differences in the internal composition of fruit from trees amply supplied with moisture compared with fruit from trees growing under conditions of moisture stress during the earlier stages of development could not be caused by a dilution factor.

Benton (1941, 1944), in Australia, advised against the use of excessive amounts of irrigation.

Koo and Sites (1955) , in their research on soil-moisture–plant relationships, determined the amount of water a mature grapefruit grove would use during a 15-month period, and classified the remaining water loss that was not directly related to the growth and metabolism of the trees. They reported that 58 per cent of the soil moisture was removed through tree transpiration, 29 per cent through percolation, and 13 per cent was lost through evaporation from the soil surface. The total quantity of water used in the experiment amounted to 65.4 acre-inches, derived from both rainfall and irrigation. (An acre-inch equals 3,630 cubic feet or 102.8 cubic meters.)

As would be expected, the amount of water transpired varied significantly with the time of year. The mean daily transpiration for the whole year amounted to 34.2 gallons (about 129 liters) per tree per day, with a range

varying from 16 gallons (60 liters) per day for February, 1952, to 53 gallons (200 liters) for June. The changes in the rates of transpiration were highly correlated with air temperature ($r = 0.834$).

The readily available moisture in the soil influences the rate of transpiration only during periods of high moisture tension. Koo (1969) noted that he found the rates of transpiration to be significant only when one-third of the available soil moisture remained in the root zone. It is not surprising, therefore, that there was found a low correlation between the rate of transpiration and the readily available soil moisture ($r = 0.062$). Since these 2 variables had some effect on transpiration, the multiple correlation coefficient ($r = 0.835$) measured the combined relationship between the dependent variable (transpiration) and the other 2 independent soil-moisture variables.

Koo (1969), in his studies on the timing of irrigation on Florida sandy soils with a low water-holding capacity, found a highly significant correlation ($r = 0.892$) between the mean monthly temperature and the average daily water loss through transpiration (figure 90). The rates of transpiration were significantly reduced at low moisture levels, irrespective of the atmospheric temperatures. The data in figure 91 show the daily evapotranspiration losses

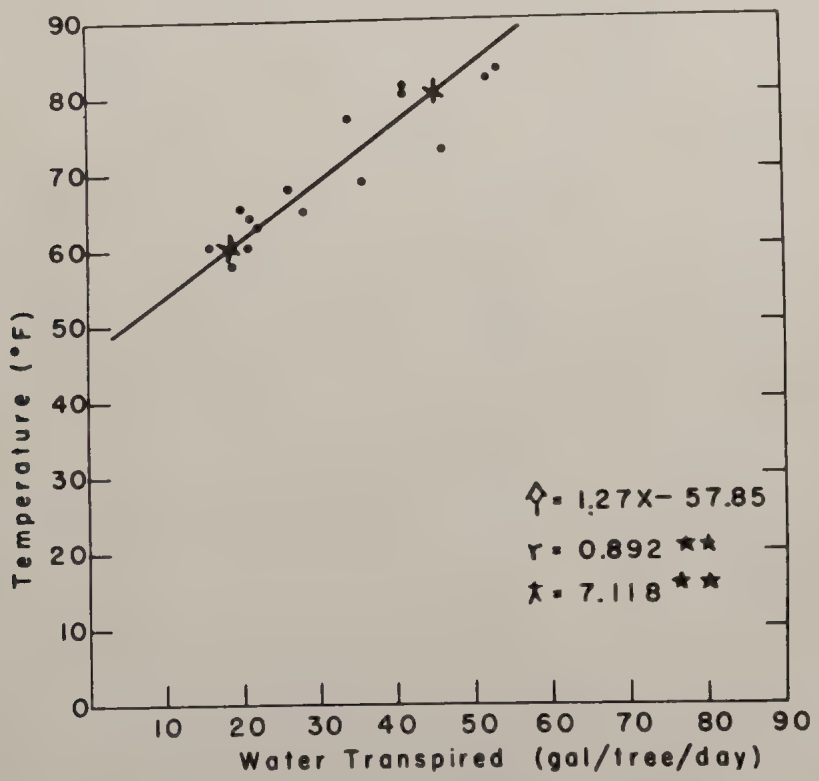


Fig. 90. Regression of mean monthly temperatures on rates of transpiration. $\hat{Y}$ equals regression estimate; r, correlation coefficient; t, test for significance. (From Koo, 1969.)

at different temperatures for mature Marsh grapefruit trees. Water consumption was calculated from the evapotranspiration data, and a valid association was observed between the calculated evapotranspiration, irrigation frequency, and fruit production. In other words, fruit production was significantly lower in groves that demonstrated a deficit in water balance resulting from evapotranspiration in excess of rainfall and irrigation.

The fruit yield depended to a considerable degree upon the available water supply, and differences in varietal responses to various irrigation treatments were observed. Varieties that responded to added water supply by increased yields also showed increased tree growth. Marsh grapefruit produced the greatest increase in fruit yield as well as in trunk growth. Under Florida conditions, Marsh grapefruit and Hamlin orange, followed by Valencia orange, showed the highest responses to irrigation, while Pineapple orange not only showed the least response in fruit yield, but produced the smallest amount of tree growth. [*Turn to page 330.*]

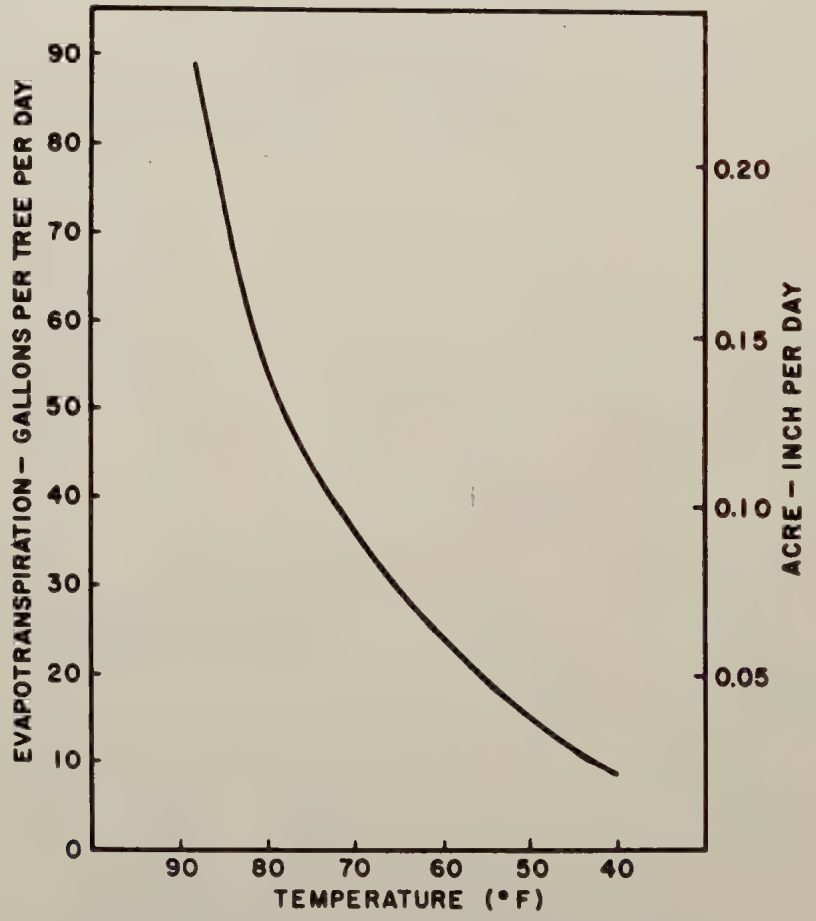


Fig. 91. Average daily evapotranspiration losses at different temperatures for mature Marsh grapefruit trees. (From Koo, 1969.)

TABLE 139

THE EFFECTS OF 3 DIFFERENT ROOTSTOCKS ON THE PALATABILITY OF GRAPEFRUIT IN 2 AREAS OF NEW SOUTH WALES.*
(Rough lemon, sour orange, and trifoliate orange.)

Areas and years	Whole fruit (specific gravity)			Juice (per cent)			Soluble solids (per cent)			Acid†			Ratios		
	R.L.	S.O.	Trif.	R.L.	S.O.	Trif.	R.L.	S.O.	Trif.	R.L.	S.O.	Trif.	R.L.	S.O.	Trif.
Leeton:															
1940	0.709	0.717	0.743	39.8	37.7	43.9	10.5	11.4	11.3	34.9	34.3	34.0	5.2	5.5	5.4
1941	.695	.678	.695	39.2	32.2	37.8	10.6	11.5	12.4	34.1	34.1	33.7	5.0	5.3	6.0
Grafton:															
1940	.816	.817	.819	47.8	50.3	40.7	9.7	9.6	10.5	30.4	28.4	29.0	5.2	5.5	5.9
1941	.720	....	.746	42.3	....	44.3	7.7	...	8.8	28.2	....	28.6	4.4	...	5.0

*From Hall (1943).
†Milliliters $N/10$ sodium hydroxide per 10 milliliters of juice.

TABLE 140

PERCENTAGES OF TOTAL SOLUBLE SOLIDS, TOTAL ACIDS, TOTAL SUGARS, AND REDUCING SUGARS IN THE JUICE OF MARSH GRAPEFRUIT FROM ROOTSTOCK PLOTS AT RIVERSIDE AND BRAWLEY (CALIFORNIA) ON VARIOUS SAMPLING DATES.*

Rootstocks	RIVERSIDE									
	May 19, 1938					May 16, 1940				
	Total soluble solids	Total acids	Total sugars	Reducing sugars		Total soluble solids	Total acids	Total sugars	Reducing sugars	
				Percentages of fresh weight	Percentages of total sugars				Percentages of fresh weight	Percentages of total sugars
Oroville sweet orange	10.7	1.83	7.49	5.08	67.8	10.5	1.50	7.50	5.64	75.2
CES 362 sweet orange	11.3	1.89	8.15	5.40	66.3	12.0	1.67	8.43	6.32	75.0
Rubidoux sour orange	11.7	1.94	8.43	5.57	66.1	11.2	1.61	7.82	6.12	78.3
African sour orange	11.1	1.89	7.80	5.43	69.6	11.9	1.62	8.21	6.25	76.1
Brazilian sour orange	11.2	1.87	8.18	5.33	65.2	12.0	1.63	8.43	6.36	75.4
Duncan grapefruit	11.0	1.76	7.88	5.43	68.9	11.4	1.57	8.00	5.47	68.4
Tresca grapefruit†	10.4	1.80	7.57	5.12	67.6	10.5	1.61	7.65	5.44	71.1
Savage citrange	12.0	1.88	8.50	5.74	67.5	12.8	1.65	9.00	6.62	73.6
Cleopatra mandarin	10.5	1.83	7.49	5.05	67.4	11.2	1.57	7.85	5.78	73.6
Sampson tangelo	11.1	1.89	7.70	5.01	65.1	11.5	1.66	8.14	5.73	70.4
Palestine sweet lime	9.2	1.54	6.54	4.56	69.7	9.2	1.37	6.24	5.02	80.4
Rough lemon	9.8	1.65	7.05	4.79	67.9	10.4	1.50	7.12	5.30	74.4
Trifoliate orange	11.9	1.87	8.73	5.87	67.2	12.5	1.63	8.92	6.38	71.5
Means	10.9	1.82	7.81	5.26	67.4	11.3	1.58	7.95	5.88	74.1

Rootstocks	November 8, 1938					November 8, 1939				
	Total soluble solids	Total acids	Total sugars	Reducing sugars		Total soluble solids	Total acids	Total sugars	Reducing sugars	
				Percentages of fresh weight	Percentages of total sugars				Percentages of fresh weight	Percentages of total sugars
Oroville sweet orange	10.6	1.70	7.80	4.23	54.2	9.5	1.65	6.84	3.91	57.2
CES 362 sweet orange	11.6	1.79	8.37	4.76	56.9	10.3	1.89	6.97	4.32	62.0
Rubidoux sour orange	11.1	1.74	8.08	4.56	56.4	10.0	1.87	7.51	3.98	53.0
African sour orange	11.0	1.74	8.16	4.02	49.3	10.2	1.74	7.35	4.17	56.7
Brazilian sour orange	11.2	1.83	8.00	4.59	57.4	10.3	1.73	7.43	4.52	60.8
Duncan grapefruit	11.2	1.83	8.46	4.68	55.3	10.0	1.67	6.98	4.02	57.6
Savage citrange	12.0	1.76	8.88	4.82	54.3	10.3	1.67	7.65	4.47	58.4
Cleopatra mandarin	11.4	1.85	8.08	4.63	57.3	9.8	1.82	7.14	4.18	58.5
Sampson tangelo	10.5	1.68	7.57	4.13	54.6	9.4	1.62	6.54	3.92	59.9
Rough lemon	9.8	1.66	7.17	4.43	61.8	9.4	1.62	6.69	4.05	60.5
Trifoliate orange	11.4	1.81	8.23	4.23	51.4	9.5	1.55	7.15	3.95	55.2
Means	11.1	1.76	8.07	4.46	55.4	9.9	1.71	7.11	4.14	58.2

*From Sinclair and Bartholomew (1944).
†The Tresca variety has sometimes been regarded as a pummelo, or at least as an intermediate type. (See Webber and Batchelor, 1943, p. 589.)

TABLE 141

PERCENTAGES OF DRY MATTER, TOTAL ASH, AND INORGANIC CONSTITUENTS OF PEEL, PULP, AND JUICE OF MARSH GRAPEFRUIT GROWN ON DIFFERENT ROOTSTOCKS AT RIVERSIDE AND BRAWLEY (CALIFORNIA).*

Rootstocks	Dry matter (fresh weight)		Total ash (dry weight)		Inorganic constituents (dry weight)											
					Ca		Mg		K		Na		SO_4		PO_4	
	River-side	Braw-ley	River-side	Braw-ley	River-side	Braw-ley	River-side	Braw-ley	River-side	Braw-ley	River-side	Braw-ley	River-side	Braw-ley	River-side	Braw-ley
PEEL																
Oroville sweet orange	18.74	25.57	3.39	5.49	0.63	1.12	0.10	0.12	0.80	1.08	0.03	0.02	0.21	0.23	0.22	0.19
CES 362 sweet orange	19.70	26.02	3.45	5.53	.66	1.17	.09	.13	.74	1.08	.03	.02	.24	.28	.24	.21
Rubidoux sour orange	19.93	26.80	3.20	5.65	.66	1.22	.10	.14	.64	1.03	.02	.02	.19	.27	.20	.20
African sour orange	19.54	28.05	3.28	5.64	.64	1.19	.11	.13	.69	0.92	.03	.03	.23	.28	.22	.19
Brazilian sour orange	20.70	26.28	3.22	5.59	.62	1.27	.09	.14	.63	0.98	.03	.03	.22	.25	.22	.20
Duncan grapefruit	20.08	26.01	3.67	5.74	.67	1.08	.10	.13	.86	1.31	.03	.02	.24	.28	.21	.18
Tresca grapefruit†	18.44	.....	3.36	....	.62	...	.09	...	.75	....	.03	...	.22	...	.23	...
Savage citrange	21.94	27.15	3.55	5.87	.70	1.17	.12	.14	.71	1.22	.04	.03	.22	.25	.25	.23
Cleopatra mandarin	19.12	27.05	3.19	5.62	.59	1.18	.09	.12	.58	0.96	.03	.03	.22	.30	.26	.18
Sampson tangelo	20.94	25.60	3.84	5.83	.68	1.21	.13	.14	.83	1.12	.03	.03	.23	.25	.24	.18
Palestine sweet lime	16.97	.....	3.10	....	.63	....	.08	...	.66	....	.03	...	.21	...	.21	...
Rough lemon	17.79	23.81	3.06	5.26	.61	1.14	.10	.12	.62	0.95	.03	.04	.22	.19	.22	.16
Trifoliate orange	21.89	27.38	3.62	5.34	.65	0.90	.12	.15	.88	1.39	.03	.03	.25	.26	.24	.25
Means	19.68	26.34	3.38	5.60	0.64	1.15	0.10	0.13	0.72	1.09	0.03	0.03	0.22	0.26	0.23	0.20

PULP

Oroville sweet orange	12.19	13.22	3.06	3.64	0.25	0.27	0.10	0.08	1.12	1.45	0.05	0.04	0.29	0.27	0.46	0.41
CES 362 sweet orange	12.61	13.87	2.96	3.70	.24	.26	.10	.09	1.08	1.53	.05	.04	.31	.26	.51	.44
Rubidoux sour orange	13.04	13.52	2.77	3.54	.26	.26	.09	.08	0.98	1.46	.05	.04	.29	.28	.44	.42
African sour orange	12.97	13.85	2.90	3.56	.26	.27	.10	.08	0.99	1.47	.05	.04	.30	.28	.45	.41
Brazilian sour orange	12.99	13.85	2.91	3.51	.28	.27	.09	.08	1.01	1.15	.05	.04	.28	.29	.45	.40
Duncan grapefruit	13.02	13.87	3.20	3.76	.23	.25	.10	.08	1.13	1.59	.05	.04	.33	.26	.48	.42
Tresca grapefruit†	12.30	.....	2.92	....	.23	...	.10	...	1.06	.....	.05	...	.31	...	.48	...
Savage citrange	14.25	14.57	3.10	3.53	.29	.24	.10	.08	1.07	1.42	.05	.04	.30	.28	.48	.43
Cleopatra mandarin	12.16	13.31	2.83	3.71	.23	.28	.11	.08	0.97	1.45	.06	.05	.31	.26	.53	.42
Sampson tangelo	13.51	12.88	3.36	3.96	.26	.28	.10	.09	1.24	1.65	.05	.05	.31	.31	.51	.44
Palestine sweet lime	10.68	.....	2.99	....	.29	...	.10	...	1.03	....	.05	...	.32	...	.52	...
Rough lemon	11.53	11.27	2.98	3.52	.27	.27	.10	.09	0.98	1.41	.05	.05	.35	.26	.50	.43
Trifoliate orange	14.38	14.64	2.99	3.83	.24	.24	.09	.09	1.00	1.60	.05	.05	.27	.29	.48	.45
Means	12.74	13.53	3.00	3.66	0.26	0.26	0.10	0.08	1.05	1.47	0.05	0.04	0.31	0.28	0.48	0.42

JUICE

Oroville sweet orange	10.58	10.56	2.55	3.43	0.07	0.07	0.11	0.11	1.15	1.26	0.07	0.06	0.20	0.19	0.37	0.33
CES 362 sweet orange	11.31	11.57	2.53	3.21	.06	.07	.11	.10	1.14	1.44	.07	.05	.22	.20	.42	.34
Rubidoux sour orange	11.71	11.11	2.71	3.26	.08	.08	.11	.10	1.19	1.37	.08	.05	.20	.19	.37	.34
African sour orange	11.11	11.03	2.52	3.22	.07	.08	.11	.10	1.16	1.48	.06	.06	.22	.20	.38	.34
Brazilian sour orange	11.24	11.17	2.58	3.25	.07	.08	.11	.11	1.18	1.16	.07	.05	.20	.22	.37	.35
Duncan grapefruit	11.04	11.17	2.76	3.54	.07	.07	.11	.11	1.30	1.60	.07	.05	.20	.22	.39	.37
Tresca grapefruit†	10.44	.....	3.03	....	.08	...	.12	...	1.27	....	.10	...	.25	...	.44	...
Savage citrange	11.96	11.97	2.66	3.16	.08	.07	.10	.10	1.22	1.38	.07	.05	.23	.20	.45	.34
Cleopatra mandarin	10.51	11.37	2.64	3.23	.07	.08	.12	.11	1.06	1.45	.08	.05	.22	.20	.42	.34
Sampson tangelo	11.11	10.51	2.94	3.47	.07	.08	.11	.12	1.20	1.57	.07	.06	.22	.21	.42	.35
Palestine sweet lime	9.18	.....	2.62	....	.08	...	.12	...	1.17	....	.07	...	.23	...	.41	...
Rough lemon	9.84	9.76	2.46	3.06	.08	.07	.12	.11	0.93	1.37	.10	.06	.25	.17	.39	.30
Trifoliate orange	11.91	11.37	2.78	3.55	.06	.07	.11	.13	1.32	1.60	.06	.06	.19	.21	.43	.35
Means	10.92	11.05	2.68	3.31	0.07	0.07	0.11	0.11	1.18	1.43	0.07	0.05	0.22	0.20	0.40	0.34

*From Sinclair and Bartholomew (1944).
†The Tresca variety has sometimes been regarded as a pummelo, or at least as an intermediate type. (See Webber and Batchelor, 1943, p. 589.)

Rootstocks. The rootstock is one of the several important factors that affect the production and composition of the fruit. This is confirmed not only by the statements and data presented in the present section, but also by other workers in Australia, South Africa, South America, the United States, and other countries who have made similar studies, not only on grapefruit but on oranges and other citrus types.

Hodgson and Eggers (1938), in California, found that trifoliate orange, the most dwarfing rootstock used, produced the highest concentrations of soluble solids in the juice of grapefruit and 5 other commercial kinds of citrus. On the other hand, rough lemon, the most vigorous rootstock, produced the lowest concentrations of soluble solids in the juice of all 6 fruits tested. Rough lemon produced the lowest concentrations of total acids and trifoliate orange the highest; this made the ratios about equal. Richards (1940), in Ceylon, obtained results similar to those of Hodgson and Eggers.

Benton (1941, 1944) expressed the opinion that, aside from delayed harvesting, improvement of the palatability of Australian grapefruit was a difficult problem because the principal factors that determined palatability were fixed by the rootstock used and by the climate—mainly temperature and humidity. However, some improvements in quality could be accomplished by limiting vegetative growth, especially during the summer and autumn.

Hall (1943) tested the effects of 3 different rootstocks in 2 areas of New South Wales. The most important results are shown in table 139. He stated that the juice of fruit grown on trifoliate orange was the least bitter, had the best flavor, and the fruit could be held on the tree longer than when grown on other rootstocks. The fruit on rough lemon matured earliest, that on trifoliate next, and that on sour orange latest.

Sinclair and Bartholomew (1944), in California, tested the effects of a relatively large number of rootstocks (13) on the composition of Marsh grapefruit. The trees were grown in 2 different areas, Riverside and Brawley. Some of the results are shown in table 140. At Riverside, the Marsh set its fruit about April 15, and at Brawley about April 1. At the time the fruits were picked, they had been on the trees for 13 months at Riverside and only 7 months at Brawley. Of the Riverside samples, those from Palestine sweet-lime stock had the lowest concentrations of total soluble solids, total sugars, and reducing sugars; those from rough-lemon stock had next to the lowest. Samples from Oroville sweet-orange and Tresca grapefruit stocks were also low in soluble constituents. Savage citrange stock yielded fruit with the highest percentage of soluble constituents, while trifoliate orange produced fruit with only slightly less. Samplings from the Brawley plots showed that rough lemon gave the lowest percentage of total soluble solids for both years, and Savage citrange the highest. Tresca pummelo (shaddock) and Palestine sweet-lime stocks were not planted in the Brawley plots.

As shown in table 140, in both years the invert sugars formed a higher percentage of the total sugars in the samples from Riverside (67.4 per cent in 1938 and 74.1 per cent in 1940) than in those from Brawley (55.4 per cent in 1938 and 58.2 per cent in 1939). These results were in agreement with those of Hilgeman and Smith (1940), who found that the greater the relative percentage of reducing sugars, the longer the fruit would hang on the tree, and the longer would be the storage life of the fruit.

Table 141 shows the relative amounts of dry matter and inorganic substances in the peel, pulp, and juice of Marsh grapefruit when grown in different areas and on 13 different rootstocks. The data show that the mean dry matter and total ash were significantly higher in the peel and pulp of fruit samples from Brawley than in those from Riverside. The mean dry-matter content of the juice was about the same in samples from the 2 areas, but the total ash was much higher in the juice samples from the Brawley plots than in those from Riverside.

Figure 92, from Sinclair and Bartholomew (1944), shows graphically the effects of such factors as locality and soil types on the relative amounts of certain soluble constituents in the juice of Marsh grapefruit grown on 11 different rootstocks.

In discussing differences in the internal quality of citrus fruits, Webber (1943) stated that the total available heat units, Riverside (3,209) and Brawley (6,078), along with the implied amount of sunshine, were more important in determining the growth rates and times of ripening of citrus fruits than other factors, such as soil types or rootstocks.

The Riverside soils in the groves represented in figure 92 belong to the Ramona series: acid for about 6 inches (15 centimeters) and alkaline below that depth. The soils in Imperial County (Brawley plots) belong to the Holtville series, and are strictly alkaline.

The statement by Webber concerning heat units should at least be taken into consideration when evaluating statements in other sections of this chapter where the effects of such factors as rootstock, soil type, and locality are discussed in relation to internal fruit quality.

Harding and Fisher (1945), in Florida, reported finding that the Marsh and Duncan grapefruit varieties were about equal in flavor when grown on sour orange, but that Duncan was found to be better than Marsh when both were on rough lemon. The fruit on sour orange contained more ascorbic acid than it did when grown on rough lemon. Total acids were found to be higher in fruit from sour orange than in that from rough lemon. Variation among groves was found to be comparatively small. The effect of rootstock on pH is shown in table 42 of chapter 2.

Harding (1947*a*, *b*) noted that grapefruit on rough lemon usually became larger, came into bearing sooner, and thus had a greater yield than trees on

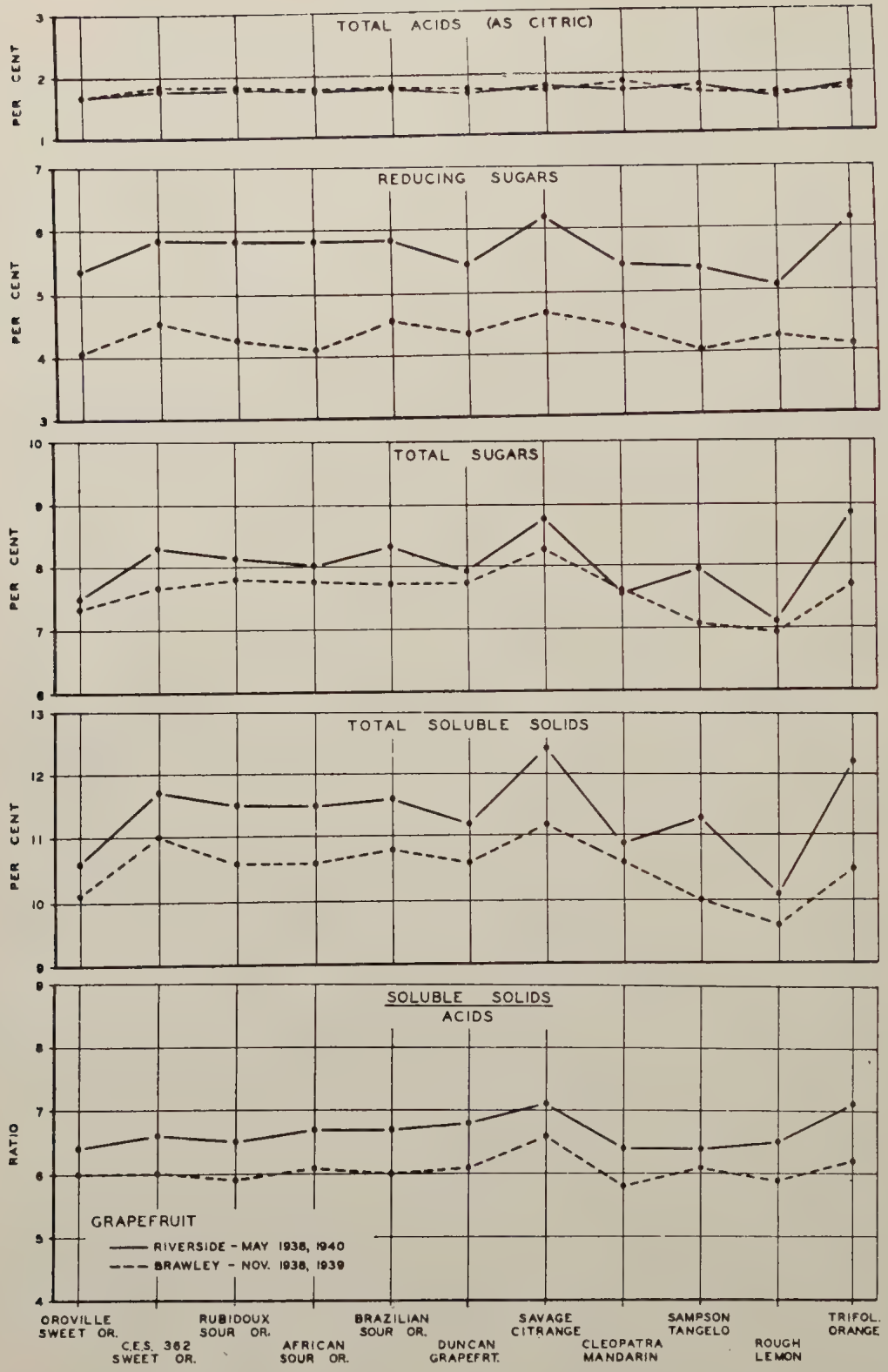


Fig. 92. Relative concentrations of certain soluble organic constituents in the juice of mature grapefruit on 11 different rootstocks, grown in plots at Riverside and Brawley, California. The mean value for 2 years is shown for each rootstock. (From Sinclair and Bartholomew, 1944.)

sour orange. Fruit grown on rough lemon averaged slightly larger, had a thicker peel, was coarser, and on a weight basis contained somewhat less juice. The eating quality was also not quite so high as for fruit grown on sour orange. The fruit from trees on sour-orange stock contained higher amounts of total soluble solids and total acids than that grown on rough lemon. Harding presented the data shown in table 142 to show the effects of the rootstock on the solids and acids in the juice. The juice from Marsh on sour orange and from Duncan on both rootstocks was acceptable for consumption by December 18; that from Marsh on rough lemon was acceptable by January 15.

Seasonal changes in the total, reducing, and nonreducing sugars of Marsh and Duncan grapefruit, as influenced by rough-lemon and sour-orange rootstocks, are shown in figure 93, from Harding and Fisher (1945). As a generalization, the rootstock will have a significant effect on the soluble carbohydrates of grapefruit. Fruit of both the Duncan and Marsh varieties contained more total, reducing, and nonreducing sugars when the trees were grown on sour orange than when grown on rough lemon.

Haas (1948) determined the relative amounts of calcium, magnesium, potassium, and phosphorus in the dry matter of the peel of Marsh grapefruit as influenced by the rootstock. Trifoliate orange rootstocks were conspicuous for their lack of agreement. Following are the 2 highest and 2 lowest values for each of 4 inorganic substances in grapefruit, from a total of 5 different rootstocks, variously distributed.

Calcium (*per cent*)		Potassium (*per cent*)	
Two highest		Two highest	
Trifoliate orange	0.637	Camulos grapefruit	0.881
Homosassa sweet orange	0.587	Lemon shaddock	0.833
Two lowest		Two lowest	
Trifoliate orange	0.472	Trifoliate orange	0.497
Lemon shaddock	0.430	Rough lemon	0.444
Magnesium (*per cent*)		**Total Phosphorus (*ppm*)**	
Two highest		Two highest	
Homosassa sweet orange	0.099	Homosassa sweet orange	850
Trifoliate orange	0.098	Camulos grapefruit	840
Two lowest		Two lowest	
Trifoliate orange	0.072	Lemon shaddock	650
Rough lemon	0.069	Trifoliate orange	620

The effects of rootstock on freeze injury are briefly discussed on page 344.

For a more complete discussion of the rootstock problems, see Batchelor and Webber (1948), and especially Webber (1948). See also, Bitters (1961).

Cohen and Reitz (1963) made an interesting study of a long-term rootstock trial for Valencia oranges and Ruby Red grapefruit. The experimental plots provided a comparison of the reactions to similar rootstocks on 2 different

soil types in areas about 200 feet (60 meters) apart. Each of the 4 experimental blocks, planted in 1950 at the Indian River (Florida) Field Laboratory, consisted of 2 plantings of Valencia and 2 of Ruby Red. One plot of each variety was planted on Leon soil, an acid soil over a layer of organic hardpan; the other was planted on Parkwood soil, a relatively fine-textured soil of near-neutral pH, underlain by marl. (*See page 336.*)

The data reported in table 143 are confined to the results on Ruby Red grapefruit. Trees of this variety on rough-lemon rootstock and Parkwood soil had the highest cumulative yield, with a total of 38.8 boxes of fruit—about 10 boxes more than trees on Cleopatra mandarin, Parson Brown, and sour-orange stocks. On the Leon soil plots, the trees on rough lemon also produced the highest cumulative yield (23.6 boxes per tree). Trees on Rangpur lime, all infected with exocortis virus and all stunted on both soil types, not only produced approximately the same cumulative yields on both soils, but had the next-to-lowest yield on Parkwood and next-to-highest on Leon.

The relative effects of rootstocks on the juice composition of Ruby Red grapefruit can also be noted from the data in table 143. The highest soluble-solids content was determined in fruit samples from sour-orange, trifoliate orange, Parson Brown, and Rangpur lime stocks; the intermediate and lowest values were obtained in samples from Cleopatra mandarin and rough lemon, respectively. Fruit from rough lemon not only had the lowest percentage of soluble solids, but also the lowest acid content. Consequently, fruit from this stock met the maturity ratio of soluble solids to acids at the times of sampling during the 7-year experimental period.

In order to assess the maturity standards for early harvest of seedless pink and Ruby Red grapefruit, Deszyck and Ting (1956) reported the seasonal

TABLE 142

SOLIDS AND ACID CONTENT OF THE JUICE OF MARSH AND DUNCAN GRAPEFRUIT ON 2 ROOTSTOCKS.*

(Averages of 4 seasons.)

Varieties and rootstocks	Soluble solids (per cent)			Total acids (per cent)			Ratios of solids to acids, December 18†
	August	May	December	August	May	December	
Marsh:							
Sour orange...	9.6	10.0	10.32	1.7	1.02	1.33	7.77
Rough lemon..	8.5	8.6	8.91	1.8	0.99	1.22	7.30
Duncan:							
Sour orange...	9.8	11.4	11.33	1.88	1.32	1.65	6.87
Rough lemon..	8.7	10.0	10.04	1.72	1.20	1.45	6.92

*Adapted from Harding (1947*a*, *b*).
†January 15 for Marsh on rough lemon.

changes in juice volumes and Brix:acid ratios for both types. The juice content increased to approximately one-third of its total volume from September to March. In the latter part of the season, the red grapefruit surpassed the pink variety in juice content. Although juice volume of the white varieties of grapefruit was apparently not affected by rootstock (Harding and Fisher, 1945), significantly more juice was found in fruit of the pink and red varie-

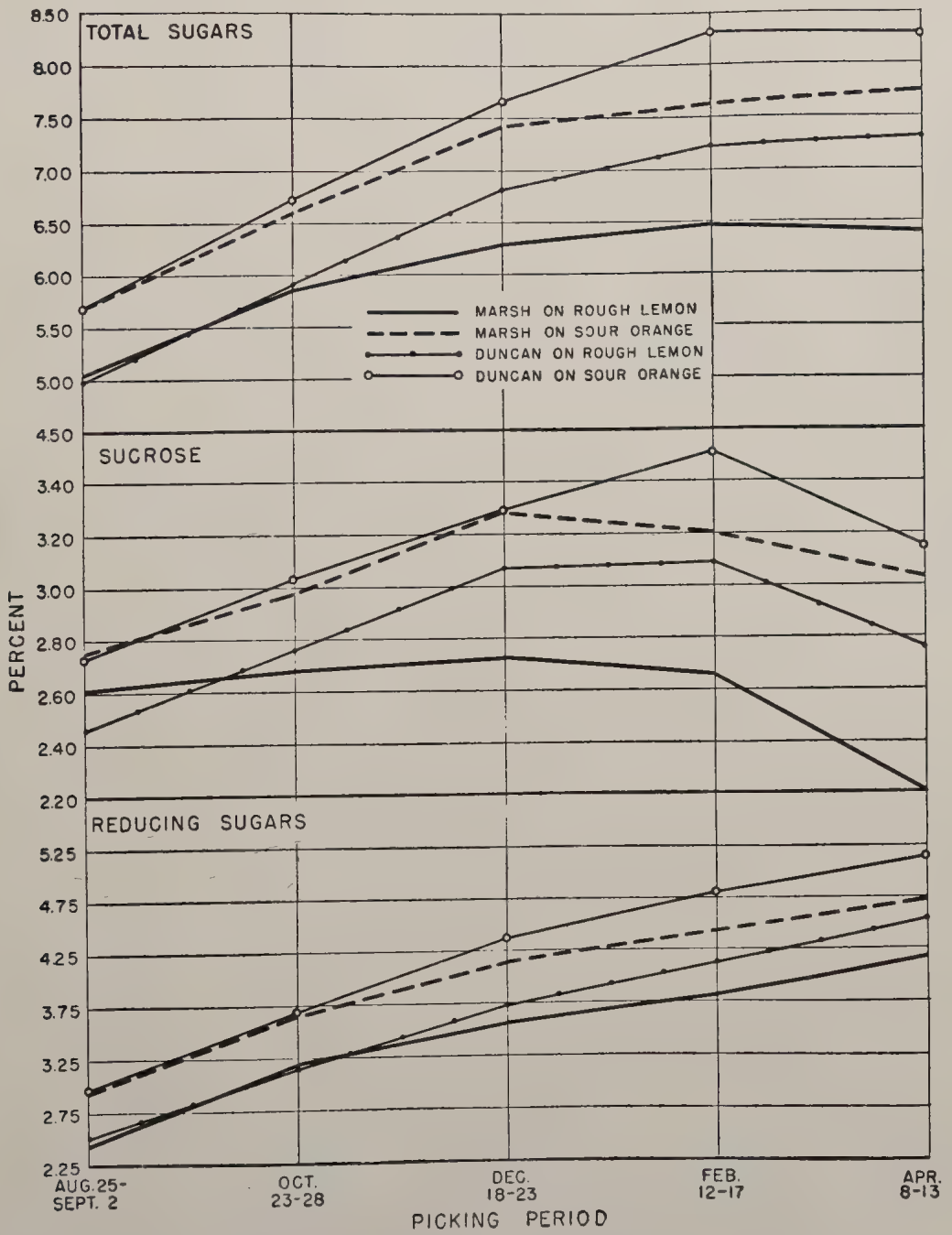


Fig. 93. Seasonal changes in total sugars, sucrose, and reducing sugars of Marsh and Duncan on 2 rootstocks, 1939–42. (From Harding and Fisher, 1945.)

TABLE 143

SUMMARY OF FRUIT QUALITY, YIELD, AND GROWTH OF RUBY RED GRAPEFRUIT, GROWN ON PARKWOOD AND LEON SOILS AT THE INDIAN RIVER (FLORIDA) FIELD LABORATORY, TO AND INCLUDING THE 1962–63 SEASON.*

(The 7 citrus types represent the rootstocks employed.)

Factors	Cleopatra		Parson Brown		Rangpur lime§		Rough lemon		Sour orange		Trifoliate§		Palestine sweet lime
	P	L	P	L	P	L	P	L	P	L	P	L	L
Average weight per fruit (grams)†	397.0	394.4	397.2	441.4	374.8	446.4	449.2	466.4	367.6	446.7	389.7	364.2	439.7
Total solids (per cent)†	8.9	9.2	9.2	9.4	9.3	9.1	8.1	8.2	9.4	9.6	9.4	9.5	9.1
Acid (per cent)†	1.14	1.16	1.15	1.16	1.12	1.10	1.05	1.07	1.15	1.17	1.13	1.10	1.11
Ratios of solids to acid†	7.9	7.9	8.0	8.2	8.3	8.2	7.7	7.7	8.3	8.3	8.5	8.8	8.2
Juice (per cent)†	49.0	49.8	48.1	49.6	49.0	51.6	49.1	52.5	48.5	51.3	49.3	47.6	50.3
Fruit yield (boxes per tree)‡	27.5	13.5	29.6	19.1	20.0	19.3	38.8	23.6	27.8	15.9	8.8	2.3	14.5
Solids (pounds per tree)‡	103.1	53.4	112.7	75.1	78.7	79.5	130.4	87.9	108.4	66.2	34.6	8.9	54.3
Average trunk circumferences, April, 1963 (inches and centimeters)	27.1	22.5	24.5	23.0	18.9	18.3	30.4	23.8	24.1	19.1	14.3	8.7	15.4
	69.0	57.0	62.0	58.5	48.0	46.5	77.0	60.5	61.0	48.5	36.2	22.0	39.0

*From Cohen and Reitz (1963).
†Seven-year averages to 1962–63.
‡Cumulative to 1962-63. Picking dates varied from February to June. (One pound equals 0.454 kilogram.)
§All trees had exocortis virus.

ties grown on sour orange than on rough lemon. The juice content was highest in red grapefruit from sour orange, and the smallest amount occurred in the pink variety on rough lemon.

In early fruit, Deszyck and Sites (1955) noted that juice volume was the maturity factor that failed to meet the requirement, and not the Brix:acid ratio; it was the ratio that was raised by maturity sprays, not the juice content.

Arsenical Sprays. In several countries, such as the United States, Russia, and especially the Union of South Africa, considerable work has been done to determine the effects of arsenical and other sprays on the internal quality of citrus fruits.

Gray and Ryan (1921), in California, were apparently the first to publish the fact that insecticidal sprays containing arsenic reduced the acidity of orange fruits. However, they quoted H. J. Webber and W. T. Swingle (unpublished) as saying that in Florida in 1893 and 1896, they had observed a reduction in the acidity of oranges sprayed with a proprietary insecticide that contained an arsenic compound and sulfur. Gray and Ryan believed that the arsenical ingredient in the spray was the substance that caused the reduction in acidity.

Several accounts follow of the results obtained by workers who have determined the effects of arsenical sprays on the quality of citrus fruits, primarily grapefruit. Longfield-Smith (1935*a*) found that the fruit from trees sprayed with 1.5 to 3 pounds of lead arsenate per 100 gallons (0.70 to 1.40 kilogram per 380 liters) of water showed a reduction in acids in the juice of 5 to 20 per cent. In some cases, the spray reduced the soluble solids and greatly hastened maturity, but made the fruit insipid and poor in quality.

Harding and Fisher (1945) stated that spraying grapefruit trees with lead arsenate in summer, at the rate of 1 pound per 100 gallons (0.454 kilogram per 380 liters) of water, was a common practice in Florida. They found that the fruit from sprayed trees was sweeter than that from unsprayed ones. The increase in sweetness was caused by the reduction in acids, which increased the solids:acids ratio in the juice (table 144). The total acid content of arsenic-sprayed immature fruit was 4 to 9 per cent below that of the unsprayed fruit, while the content of the very ripe sprayed fruit was as much as 21 to 26 per cent less. The lead arsenate spray did not significantly lower the juice volume (computed as milliliters per 100 grams of fruit), the concentration of total soluble solids, or the concentration of ascorbic acid, but it did result in a slight decrease in the size and weight of the fruit.

To determine the effect of arsenical sprays on the composition and quality of grapefruit, Rygg and Getty (1955) repeated the Florida experiments of Harding and Fisher, using fruit grown in Arizona and California. They found that fruit from arsenical-sprayed trees yielded slightly higher values for juice content, total sugars, ratio of soluble solids to acid, and palatability, but

TABLE 144

EFFECTS OF LEAD ARSENATE SPRAY ON THE AVERAGE TOTAL ACID OF GRAPEFRUIT AT DIFFERENT PICKING PERIODS, 1939–43.*
(Minus signs indicate that total acid was less in sprayed fruit than in unsprayed fruit.)

Varieties and rootstocks	Total acidity (per cent)									
	Aug. 25–Sept. 2	Sept. 25–30	Oct. 23–28	Nov. 20–25	Dec. 18–23	Jan. 15–20	Feb. 12–17	Mar. 11–16	Apr. 7–13	May 6–11
Marsh on rough lemon:										
Unsprayed	1.86	1.48	1.55	1.52	1.45	1.38	1.29	1.23	1.14	1.01
Sprayed	1.73	1.37	1.37	1.33	1.25	1.15	1.12	1.04	0.92	0.80
Differences (per cent)	−0.13	−0.11	−0.18	−0.19	−0.20	−0.23	−0.17	−0.19	−0.22	−0.21
Marsh on sour orange:										
Unsprayed	2.05	1.63	1.68	1.60	1.47	1.44	1.39	1.31	1.18	1.14
Sprayed	1.86	1.51	1.53	1.39	1.30	1,19	1.16	1.10	0.95	0.84
Differences (per cent)	−0.19	−0.12	−0.15	−0.21	−0.17	−0.25	−0.23	−0.21	−0.23	−0.30
Duncan on rough lemon:										
Unsprayed	1.81	1.55	1.62	1.61	1.56	1.52	1.47	1.44	1.32	1.23
Sprayed	1.67	1.39	1.43	1.38	1.35	1.27	1.24	1.13	1.06	0.97
Differences (per cent)	−0.14	−0.16	−0.19	−0.23	−0.21	−0.25	−0.23	−0.31	−0.26	−0.26
Duncan on sour orange:										
Unsprayed	2.06	1.83	1.97	1.97	1.93	1.81	1.78	1.68	1.63	1.53
Sprayed	1.97	1.65	1.71	1.69	1.59	1.54	1.45	1.42	1.29	1.21
Differences (per cent)	−0.09	−0.18	−0.26	−0.28	−0.34	−0.27	−0.33	−0.26	−0.34	−0.32

*From Harding and Fisher (1945).

slightly lower acid content, than that from unsprayed trees. No noticeable effects were observed on the amounts of soluble solids, reducing sugars, and ascorbic acid. They concluded from these results that the differences in composition between fruit from sprayed and unsprayed trees were insignificant, and that the experimental data did not justify the application of arsenical sprays to accelerate the maturity of grapefruit under Arizona and California conditions (figure 94).

On the other hand, Sites (1944) stated that in Florida, without the use of arsenical sprays, grapefruit juice was likely to be relatively high in acids and to have a resultant sour flavor. This was especially true of late-bloom fruit, with its short growing and maturing period, which might result in juice with a relatively high acid content. In a later paper (1947), Sites said that aside from the use of arsenical sprays, there was no short cut to the commercial maturity of grapefruit.

Harding (1944, 1946) reported that 1 summer application of lead arsenate had no marked effect on the ascorbic acid content of either Marsh or Duncan grapefruit juice.

State regulations in California prohibit the use of arsenical sprays on all kinds of commercially grown citrus (California Administrative Code, 1949).

In 1952, S. A. Trout[1] reported that arsenical sprays were seldom used in Australia on grapefruit; but when they were, they were applied early in the summer, at the rate of 2.5 pounds of lead arsenate per 90 U. S. gallons of water (1.14 kilogram per 341 liters).

For a more detailed discussion of the effects of lead arsenate and other sprays on the quality of citrus fruits, see Rose *et al.* (1951).

In contrast to the effects of arsenical sprays, Deszyck *et al.* (1952), working in Florida, found that in general, the total acid content of Duncan grapefruit juice increased after the application of copper sprays to the trees, and that the increase was evident throughout the sampling season. They also noted some indication of a cumulative effect, since the increment in acid increased slightly each season from 1949 to 1951. As would be expected, the copper sprays significantly decreased the sugar:acid ratios of the juice, and thus delayed fruit maturity. When copper and lead arsenate were sprayed on the same trees, the total acid increased and the ratios of the juice decreased, as compared with fruit from trees receiving lead arsenate alone. The results of these tests showed that lead arsenate spray alone produced an increase of 0.33 ratio unit, while the addition of copper spray reduced the effectiveness of the lead arsenate by 0.16 unit. In other words, when copper spray was added to the lead arsenate spray, the effectiveness of the latter in lowering the total acid was reduced approximately 50 per cent.

[1] S. A. Trout, Director of Horticulture, Department of Agriculture and Stock, Brisbane, Australia. Personal correspondence.

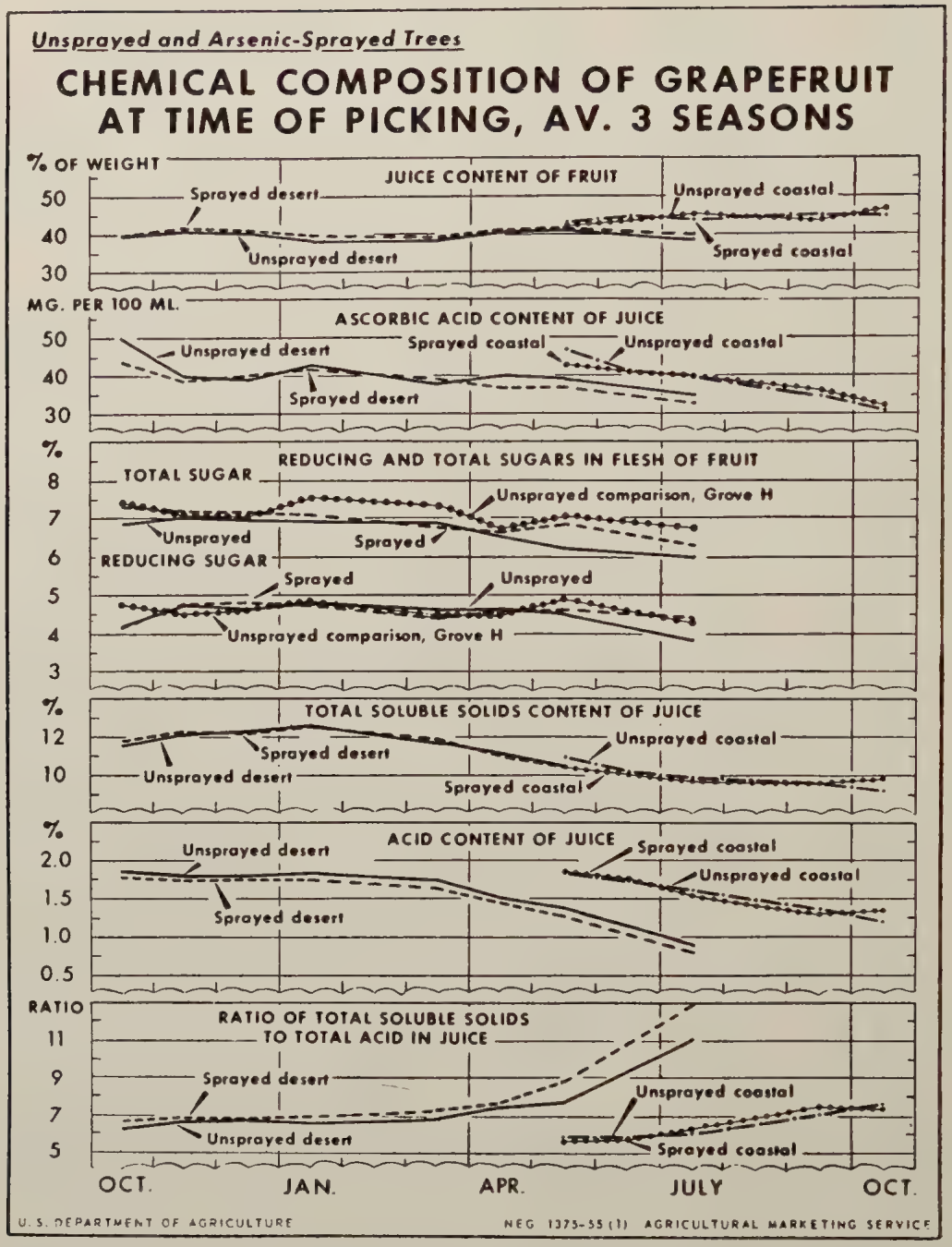


Fig. 94. Relationships of arsenical sprays to juice, ascorbic acid, reducing and total sugars, total soluble solids and acid, and total soluble solids:acid ratios in desert and coastal grapefruit. Averages for 3 seasons. (From Rygg and Getty, 1955.)

Deszyck and Sites (1955) determined the juice contents and ratios of Brix to acids in Ruby Red grapefruit for a 5-season period. The juice content increased with maturity, varied with the season, and was not influenced significantly by foliar sprays of lead arsenate of 0.4 to 1.6 pound per 100 gallons of water (0.18 to 0.73 kilogram per 380 liters). A foliar spray of lead arsenate at 0.6 pound (0.27 kilogram) per 100 gallons was recommended for early maturity of the Ruby Red variety.

In another report, Deszyck and Ting (1958) presented data on the seasonal changes in acid content of Ruby Red grapefruit sprayed with different amounts of lead arsenate in 1 orchard for 6 seasons (table 145). The acid content varied annually between 1.23 and 1.68 per cent. The first 3 seasons (1951–1954), higher rainfall produced fruit with lower acid contents in the juice. The inverse relationship between mean acid content and rainfall for the entire period (August through January) was demonstrated by the highly significant negative correlation coefficient ($r = -0.737$). Although the effect of lead arsenate on grapefruit was markedly influenced by seasonal conditions, the loss of acids in fruit sprayed with the highest concentration was more than twice the loss in fruit treated with the lowest concentration.

TABLE 145

EFFECTS OF LEAD ARSENATE SPRAY AND TOTAL RAINFALL DURING SAMPLING PERIODS ON THE ACID CONTENT OF RUBY RED GRAPEFRUIT IN 1 ORCHARD FOR 6 SEASONS.*
(Means of triplicate plots on 5 sampling dates.)

Crop seasons	Rainfall†		Lead arsenate (pounds per 100 gallons of spray)‡					
	Inches	Centimeters	None	0.4	0.8	1.2	1.6	Means
			Acid content (per cent)					
1951–52	28.9	73.5	1.23	1.13	1.06	1.02	1.00	1.09
1952–53	29.7	75.5	1.50	1.35	1.26	1.25	1.22	1.31
1953–54	34.9	88.7	1.30	1.11	1.01	0.92	0.95	1.06
1954–55	14.5	36.8	1.49	1.38	1.32	1.28	1.25	1.35
1955–56	12.9	32.8	1.68	1.64	1.49	1.51	1.49	1.56
1956–57	22.5	57.2	1.46	1.41	1.37	....	....	1.41
Means	....	....	1.44	1.34	1.25	1.20	1.18	....

Rates and seasons:
Least significant difference at the 5 per cent level.....03
Least significant difference at the 1 per cent level.....04
Interactions: Rates × seasons:
Least significant difference at the 5 per cent level.....04
Correlation coefficient between rainfall and rates: −.737**.
r figured at 5 per cent: −.374.
r figured at 1 per cent: −.478.

*From Deszyck and Ting (1958).
†Rainfall during August through January.
‡One pound equals 0.454 kilogram; 100 gallons equal 380 liters.

Although arsenical sprays were being used in Florida for the specific purpose of reducing the acidity of grapefruit juice, Deszyck and Ting (1959) reported other fruit and tree characteristics that were influenced by lead arsenate sprays. The treated trees produced fewer fruits of smaller size, and dropped fewer immature and preharvest fruits than untreated trees. The influence of lead arsenate on yields varied with seasonal conditions; reductions in yields varied from slight to very pronounced losses. The sprayed fruits had slightly thicker rinds and lower ratios of equatorial:polar (E:P) diameters.

In another experiment on Marsh, Duncan, and Ruby Red grapefruit, Deszyck and Ting (1960) sprayed the trees with lead arsenate, using up to 3 pounds per 100 gallons of water (1.40 kilogram per 380 liters). The treated fruit of the 3 varieties contained less reducing sugar and more nonreducing sugar, and significantly more total sugars than untreated fruit. The so-called bioflavonoids, expressed in terms of naringin (the chief component of this group), were also significantly higher in the treated Marsh and Ruby Red fruit.

Atkins and Rouse (1958) reported that spraying Silver Cluster grapefruit trees with lead arsenate had no significant effect on the keeping quality of canned sections stored for a period of 36 weeks at 80° F. (27° C.). The water-soluble pectic substances were not changed to oxalate-soluble pectic materials at a faster rate than the hydrolysis of protopectin. According to the authors, the protopectin was slowly hydrolyzed to water-soluble pectic substances in the presence of the hydrogen ions produced by the natural acids of the sections, and this slow reaction resulted in an increase in both water-soluble and oxalate-soluble fractions, with a decrease in sodium hydroxide-soluble pectic substances.

Vines and Metcalf (1967), in searching for an explanation of why lead arsenate reduced the acid content of grapefruit, suggested that the seasonal rise and fall of citric acid in the juice might be correlated with the general phosphorylative ability of grapefruit mitochondria. In an earlier report, these authors observed the proportionality between the decrease in phosphate esterification and the increase in arsenate in grapefruit mitochondria (Metcalf and Vines, 1965). This effect was ameliorated by increasing the inorganic phosphate in the reaction medium. Since the accumulation of citric acid against a concentration gradient in the vacuole of the juice cell requires energy, a decrease in the available energy should result in a decreased capacity for transport and storage of organic acids.

Freeze Injury. The subjection of grapefruit trees to freezing and subfreezing temperatures produces injury to both trees and fruit. Freeze injury to the fruit produces various internal physical and chemical changes which result in many adverse effects on both quality and yield. The degree of freeze dam-

age is usually estimated from the exposed surface by dividing the fruit into stem, center, and stylar thirds, and thus calculating the percentages of injury on the basis of the total volume of the fruit. The symptoms that are usually observed when using a transverse cut on fruit suspected to be frost-damaged are: (1) pale or milky appearance of the cut surface; (2) mushiness of exposed pulp; (3) reduced volume of juice vesicles may indicate partial dryness; (4) wavy or "snaky" segment walls; (5) other physical symptoms, such as scattered groups of gelatinized vesicles, light-yellow grains or granules in some vesicles, gum pockets, and perhaps 2 segments formerly hollow, but now completely or partially filled in because of the excessive growth of uninjured segments; and (6) desiccation of the vesicles and segments. This last-named type of injury is the most consistent and reliable indicator used to determine internal fruit injury (Bartholomew *et al.,* 1950).

It has been observed that grapefruit subjected to freezing temperatures and picked immediately thereafter may show a delay of 10 to 20 days before developing internal injury. Under these conditions, the rate of development of freeze damage depends upon the injury sustained at the period of low-temperature exposure and other orchard variables. Some variable factors that may influence the amount of freeze injury to fruit at any given time of exposure to freezing temperatures are: (1) the degree of maturity; (2) the minimum temperatures in the grove; (3) how long the freezing temperatures have endured; (4) fruit and atmospheric temperatures preceding actual exposure to the freezing temperatures; and (5) the air velocity in the grove. These are only a few of the variable components that may affect fruit injury under low-temperature conditions. However, note that the small fruits on a tree are more likely to be frozen than the larger ones.

Camp (1935) stated that freeze damage to citrus trees and fruit was found to depend upon factors such as grove location, grove management, and methods of cold protection. The factors having to do with grove location are concerned with such things as contour of the land, air drainage, water protection, and character of soil. The data from freeze surveys seemed to support the belief that tree condition was the most important feature of the responses of citrus trees to cold weather. Healthy, vigorous trees consistently withstood the cold temperatures better than weak or unhealthy ones. Rootstock and varietal differences within a species were not important in helping the trees to withstand cold temperatures. More freeze injury occurred on trees carrying a crop, and green fruit was more easily injured than mature fruit.

The tests of Semakin and Moros (1941) showed that the resistance of mandarin orange and grapefruit to frost injury was increased by normal doses of: (1) $NaNO_3 + KH_2PO_4 + Na_2HPO_4$; (2) $KCl + NH_4NO_3$; and (3) NH_4NO_3.

The Florida Citrus Commission (1951) stated that concentrates made from frozen fruits showed more gelation than those made from nonfrozen fruits.

Hume (1926), Uphof (1938), Hodgson and Wright (1950), and Batchelor and Rounds (1948) all reported that oranges and grapefruit on rough-lemon rootstock were found to be more susceptible to cold injury than they were on sour orange.

Cooper (1952), in Texas, found that trees grown on rough lemon generally made a better recovery than those on sour orange.[2] The trees were not over 3 years old, but his conclusions were based on observations in many localities. Cooper *et al.* (1963) reported data showing that large variations in freeze injury which occurred among different rootstock combinations growing in different climates were partially explained by differences in cold-temperature requirements to induce dormancy in the scion-rootstock combinations.

After the freezes of 1948–49 in California, Bartholomew *et al.* (1950) determined the nature and extent of injuries to oranges and grapefruit. Their studies were mostly made on oranges, but the data presented here apply to grapefruit only. The extent and distribution of freeze injuries in the 2,800 fruits examined are stated as percentages.

	Fruits	*Segments*
In stem end	32.4	40.0
In center	39.4	35.2
In stylar end	28.2	24.2

Of the 32.4 per cent of fruits frozen in the stem end, 63.3 per cent were from the outside and 36.7 per cent were from the inside of the trees, while of the 39.4 per cent frozen in the center, 70.4 per cent were outside and 29.6 per cent were inside fruits. Almost all frozen grapefruit that remained on the trees from the time they were injured (early January) until they were picked (May and June) showed signs of recovery. The uninjured segments increased in size enough to fill, or nearly fill, the spaces formed by the injured segments or parts of segments. From the over-all results obtained with both oranges and grapefruit, it appears justifiable to conclude that a transverse stem-end cut as well as a transverse center cut should be made to determine the actual severity of freeze injury.

Klotz and DeWolfe (1954) described a type of injury to the surface of grapefruit which for many years had been ascribed to various factors, such as the virus of psorosis B, sprayburn, smog, and low temperatures accompanied by ice formation (figure 95). The pattern of the injury is made up of individual small pits, which coalesce in some areas to make larger ones. If the injury takes place when the fruit is green, each pit is surrounded by a green halo, which persists for a time after it has acquired the yellow color of maturity. Actually, the injury differs from that caused by the psorosis virus, in that the latter

[2] These apparently contradictory statements frequently occur in adjacent citations, and are quoted merely to show the results of the individual authors who made the investigations. Many unpredictable factors enter into such findings.

has a tendency to form rings. It is not made up of individual pits, but of depressed, continuous rings which become brown and necrotic. Although these investigators did not reproduce the blemish experimentally, the most generally accepted theory at present is that the damage occurs when the temperature is low enough to freeze free water that collects as dew or rain on the surface of the peel. The edges of the wet areas or drops freeze first, then the new boundaries of the thicker layers of retreating water freeze successively, causing the concentric circles of injury. This type of damage has been found in smog-free locations and on fruit from virus-free trees. It is most generally found in the colder parts of the orchard and on fruit exposed to the sky, which thus is most influenced by radiation.

Rouse *et al.* (1958) compared some physical and chemical properties of nonfrozen and frozen juices from Pineapple orange and seedy grapefruit that

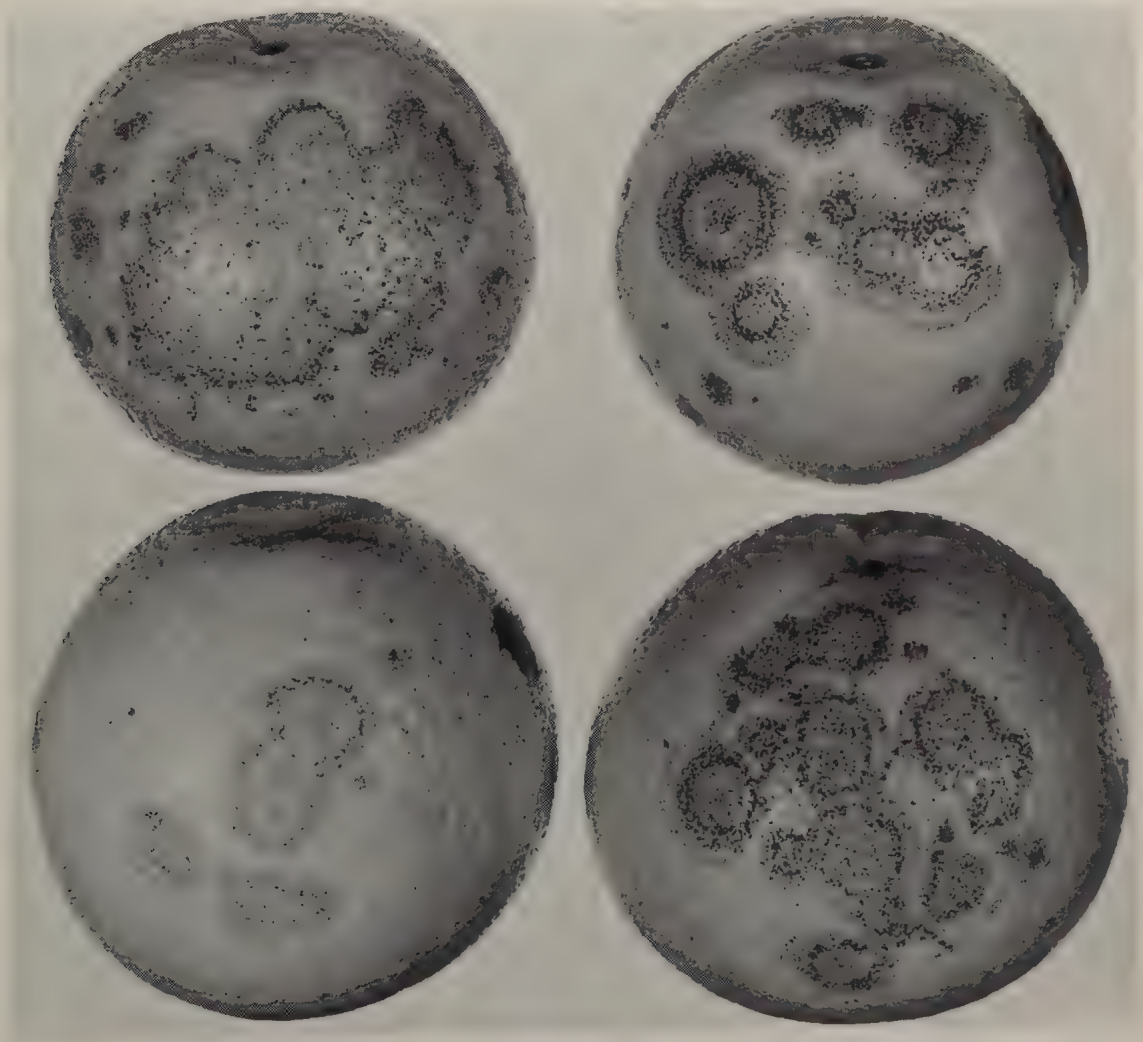


Fig. 95. Blemishes on grapefruit, sometimes attributed to temperatures low enough to freeze free water collected as dew or rain on the surface. (From Klotz and DeWolfe, 1954.)

TABLE 146

COMPOSITION CHANGES IN JUICES FROM SOUND AND FROZEN PINEAPPLE ORANGES AND SEEDY GRAPEFRUIT.*

(Hand-extracted by electric reamer. Data determined on centrifuged juices or serums.)

Factors	Pineapple oranges picked January 16, 1958			Seedy grapefruit picked February 25, 1958		
	Not damaged	Damaged by freezing	Seriously damaged by freezing	Not damaged	Damaged by freezing	Seriously damaged by freezing
Degrees Brix (spindle at 17.5° C.)	10.60	10.10	10.95	9.65	9.85	9.50
Total acid (as citric)	1.01	0.87	0.49	1.55	1.35	1.15
Brix:acid ratios	10.50	11.61	22.35	6.23	7.30	8.26
pH	3.7	3.8	4.2	3.3	3.4	3.5
Water-insoluble solids (mg per 100 grams)	235.2	206.8	206.8	64.0	71.2	87.2
(PEu)g soluble solids × 1,000	21.3	19.1	14.3	17.6	12.2	11.7
Flavonoids (mg per 100 ml)	116.1	121.9	137.8	27.5	31.0	35.7
Ascorbic acid (mg per 100 ml)	...	...	...	33.3	32.6	32.4
Light transmittance	35.2	34.1	26.9	69.5	67.0	65.5
Relative viscosity at 26° C. (79° F.)	1.92	2.13	4.39	1.31	1.43	1.57
Pectin in serum (mg per 100 grams)	41.3	43.3	56.7	15.3	16.7	19.3
Pectic fractions:						
Water-soluble (mg per 100 grams)	39.3	41.3	54.7	18.0	20.7	24.7
Ammonium oxalate-soluble (mg per 100 grams)	21.3	20.0	24.0	9.3	10.3	12.0
Sodium hydroxide-soluble (mg per 100 grams)	42.0	38.0	25.3	14.7	15.3	14.3
Totals (mg per 100 grams)	102.6	99.3	104.0	42.0	46.3	51.0
Percentages of total pectin:						
Water-soluble	38.3	41.6	52.6	42.9	44.7	48.5
Ammonium oxalate-soluble	20.8	20.1	23.1	22.1	22.2	23.5
Sodium hydroxide-soluble	40.9	38.3	24.3	35.0	33.1	28.0

*From Rouse *et al.* (1958).

had been exposed to several severe freezes in central Florida during the 1957–58 season. The Pineapple orange samples harvested on January 16, 1958, had been subjected to both the December and January freezes. The seedy grapefruit samples harvested on February 25 had been exposed to all the freezes occurring during December, January, and February. It should be noted that both oranges and grapefruit were mature when the freezes occurred. The data in table 146 show that total acidity, pectinesterase activity, and percentages of light transmission decreased in the Pineapple orange and seedy grapefruit juices with an increase of internal freeze injury to the fruit. In the order just cited, the characteristics of grapefruit juice decreased from 1.55 to 1.15 per cent, 17.6 to 11.7 units, and 69.5 to 65.5 per cent. A reduction in light transmission indicates an increase in cloud or turbidity of the juice. With the decrease in total acidity, it follows that the Brix:acid ratios and pH should increase with the severity of injury. The flavonoids (determined as hesperidin in orange and naringin in grapefruit), serum pectin, and serum viscosity increased in both the orange and grapefruit juices as the amount of freeze injury increased.

Hill and Wenzel (1963) determined some highly relevant data on the development of microorganisms in frozen citrus fruits which may be appropriately reported at this point. They compared the microbial deterioration of oranges that remained on the tree after a severe freeze with the damage to those that dropped and stayed on the ground for various lengths of time. The fruits (1,249 oranges) that were frozen, artificially or naturally, and remained on the tree yielded 1 orange having juice with a plate count of more than 1 million colonies per milliliter, and only 2 with juice having counts of over 50,000. These data seem to support the conclusion that frozen oranges left on the tree (or picked for analysis) are safe, from a microbial standpoint, for use as fresh or processed fruit. Under Florida conditions, fermentation did not occur significantly in frozen oranges, no matter how long the fruit remained on the tree after freezing.

Hill and Wenzel noted that frozen oranges that fell to the ground deteriorated very rapidly as a result of the accelerated development of stem-end rot and surface molds. The microbial populations in juice samples from dropped frozen oranges were greater in number than in similar samples from fruit picked from the tree. The general observations made on dropped fruit were based on data obtained on a total sample of 684 oranges, of which 57 were infested with microorganisms and 42 had plate counts of over 1 million. The dropped oranges stayed on the ground for 43 days, and the experimental samples were selected from fruits that had no observable damage.

In these experiments, it was found that when some oranges were pressed while submerged in water, streams of bubbles were emitted through the invisible microapertures of the flavedo. When this technique was applied to

samples from both picked and dropped fruit, bubbles were emitted from 105 dropped oranges, in contrast with only 6 that had been picked from the trees. It was suggested that these microapertures could serve as foci of infection, enabling microorganisms to enter the fruit.

The juice of freeze-damaged fruit contained significantly more water-soluble pectic materials than that from sound fruit, but the ammonium oxalate-soluble pectin increased only slightly in juice from the seriously damaged specimens. The juice of frozen grapefruit showed only a slight reduction in sodium hydroxide-soluble pectic substances, but similar alkali-soluble fractions obtained from the juice of frozen Pineapple oranges showed a significant decrease. The changes that occurred in the pectic fractions may be clearly demonstrated by expressing each fraction as a percentage of the total extractable pectic substances (table 146).

After the Florida freeze of December, 1962, Rouse *et al.* (1963) again studied the effects of subfreezing temperatures on component parts of citrus fruits, with special reference to pectic substances. The data of table 147 show that the average weight of whole Silver Cluster grapefruit decreased from 473.6

TABLE 147

REPORT ON THE CONDITION OF SILVER CLUSTER GRAPEFRUIT BEFORE AND AFTER THE FLORIDA FREEZE OF 1962.*

Part 1. Changes in whole fruit and component parts.

Months when sampled	Whole fruit		Component parts (per cent)			
	Average weight (grams)	Total solids (per cent)	Peel	Pulp	Seeds	Juice
December (control)	473.6	17.02	24.6	29.9	4.3	41.2
January	472.4	16.51	30.3	26.0	4.1	39.6
February	417.9	16.83	31.2	30.9	4.8	33.1
March	437.0	15.65	30.1	29.9	3.9	36.1
April	476.6	14.89	33.4	24.2	4.1	38.3

Part 2. Juice analyses before and after the December, 1962, freeze.

Months when sampled	Total solids (per cent)	Degrees Brix	Total acid (per cent)	Brix:acid ratios	pH
December (control)	11.15	11.91	1.73	6.88	3.33
January	10.19	10.93	1.41	7.75	3.50
February	10.42	11.26	1.47	7.66	3.45
March	10.09	10.99	1.45	7.58	3.50
April	9.62	10.51	1.31	8.02	3.55

*From Rouse *et al.* (1963).

grams (control sample) to 417.9 grams from December to February, and that in the April sample, the weight had increased to 476.6 grams. For the same sampling period, the percentages of juice followed a similar pattern, decreasing from 41.2 to 33.1 per cent, and subsequently rising to 38.3 per cent. The average total solids of the whole fruit decreased from 17.02 to 14.89 per cent from December to April. During the same period, the peel content increased from 24.6 to 33.4 per cent, and the pulp content decreased from 29.9 to 24.2 per cent.

As a result of the freeze, the time course of the pectinesterase activity of the pulp gradually increased from 125.9 units in December to 219.2 units in April. The ammonium oxalate-soluble pectin of the pulp more than doubled during the experimental period, changing from 8.1 per cent before the freeze in December to 19.4 per cent in April (figure 96). As the total acid values decreased, the Brix:acid ratios increased, with corresponding increases in pH values.

Without developing an extended treatise on cold-hardiness in citrus trees in relation to dormancy, a summary of the experimental data should be presented in order to round out this section discussing the nature of freeze damage to grapefruit.

Cold-hardiness is a characteristic common to many plants of the temperate zone. Tropical species are highly susceptible to injury at low temperatures. Although tropical and subtropical plants may be damaged by temperatures somewhat above freezing, damage at low temperatures generally results from

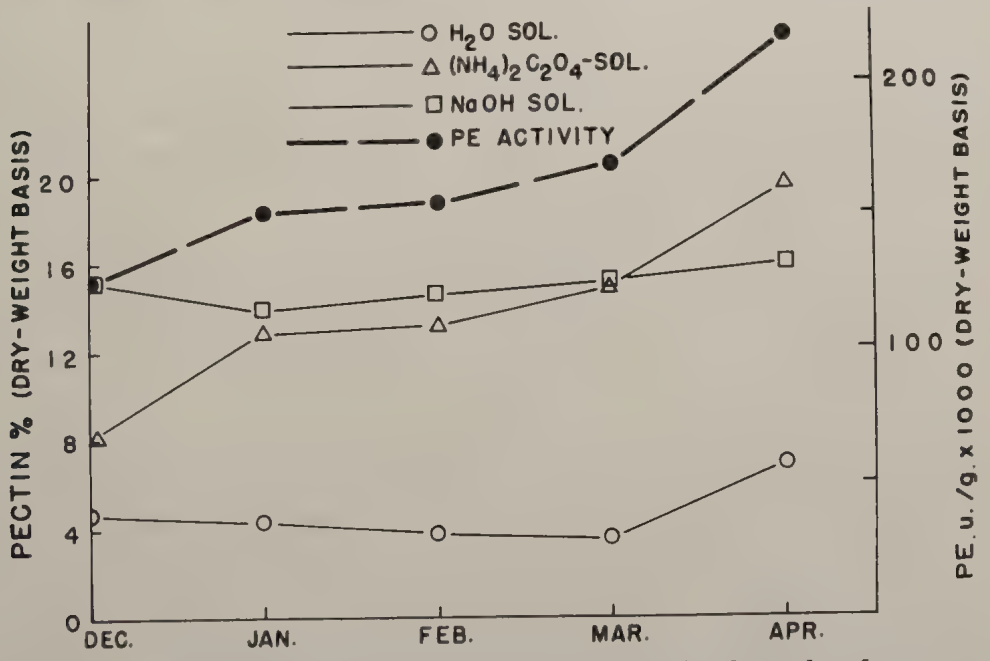


Fig. 96. Pectic fractions and pectinesterase in the pulp of Silver Cluster grapefruit before and after the Florida freeze of December, 1962. (From Rouse *et al.*, 1963.)

the freezing of tissues, which is associated with degrees of cold below the freezing point of water. Because of their hydrated characteristics, most plants freeze when exposed to temperatures slightly below 32° F. (zero centigrade). From an experimental standpoint, cold-hardiness in citrus trees is measured in terms of the degrees of cold to which dormant trees must be exposed in order to produce injury, compared with the temperatures at which non-dormant trees are injured.

Cooper and Peynado (1959) investigated cold-hardiness in citrus as related to dormancy. Cooper *et al.* (1954), using a specially constructed portable tree freezer, exposed young trees in the field to controlled freezing temperatures and compared, on a physiological basis, the degree of cold-hardiness they had attained. The data from these experiments showed that cold-hardiness of citrus trees was associated with the degree of dormancy they exhibited. Dormancy involved the cessation of growth of cambium, roots, and shoots resulting from exposure to drought and/or cold. Experimentally, the measurement of extension growth of new shoots was used as an index of bud dormancy; i.e., when no linear growth occurred, the buds were considered to be dormant. Radial growth, which was measured by increases in the areas of cross-sections of twigs, branches, and trunks, indicated the presence of cambial activity. In the absence of radial growth, the cambium was assumed to be dormant. As an index of active root growth, these investigators counted the white root tips at 2-week intervals.

Cooper *et al.* (1955), in studying the effects of drought on the dormancy and cold-hardiness of citrus trees, showed that 3-year-old Meyer lemon trees could be protected from cold by maintaining a drought condition in the soil. The trees were not irrigated between November 12, 1954, and February 12, 1955, a period during which no rain occurred and night temperatures fell below 40° F. (4.4° C.) on 12 occasions at widely different intervals. These scientists, using the tree-freezer technique, found that the dry trees were injured by exposure to 26° F. (– 3.3° C.) for 3 hours.

In studying the effects of wet and dry soil on the growth of grapefruit trees, Husain and Cooper (1958) noted that cambial activity during the winter developed more slowly in the terminal shoots of dry trees than in the shoots of wet ones.

In winter, bud dormancy is usually accompanied by cambial dormancy in both twigs and branches, but not in the trunk. As a result of dormancy-inducing temperatures, cambial activity in citrus trees is significantly reduced during the winter in Florida and Texas, and is usually stopped completely in Arizona and California.

Cooper *et al.* (1964) reported on an experiment that occurred naturally in Texas during the 1949 freeze, showing the relationship between drought and cold-hardiness. Many nurserymen had not irrigated their surplus stock for

several months before the freeze. The nonirrigated nursery trees were all uninjured, the irrigated trees in certain nurseries were uninjured, while the irrigated trees in still other nurseries were severely injured.

Young and Peynado (1961), studying changes in the cold-hardiness characteristics of 10-year-old Redblush grapefruit trees, used a large portable freezer to subject field trees to controlled temperature conditions in order to make their observations. The prevailing air temperatures in the Rio Grande Valley in the winter of 1960–61 were 65 to 70° F. (18 to 21° C.) for November, 45 to 60° (7.2 to 16° C.) for December and January, and again 65 to 70° for February and March. The physiological characteristics of the shoots and bark at these temperature ranges were borne out by the fact that during November, the shoots were dormant but barely mature and the bark was slipping throughout the tree; in December, the shoots were fully mature and the bark was slipping; in January, the bark on the shoots was tight and slipped with difficulty; and in February, the new shoots were 1 to 6 inches (2.5 to 15 centimeters) long, and the bark was succulent and slipped easily. Trees existing under these growth conditions were exposed to 23° F. (– 5° C.) for 4 hours during the third weeks in November, December, and January, and the first week in March. The freeze damage resulting from these monthly low-temperature exposures showed that leaf and twig injury was severe in November; was less severe in December; there was slight leaf injury and no twig damage in January; and there was severe leaf and twig damage in March with the approach of the spring flush of growth. These data indicate quite significantly that cambial dormancy which is correlated with cool winter weather will result in an increase in cold-hardiness.

Of the many climatic variables that influence the cold protection of citrus trees, changes in temperatures during the fall and winter are highly related to the physiological changes that influence dormancy, and consequently freeze injury, to fruits and tissues. In the citrus-growing districts of the United States, freezes usually occur in December, and the preconditioning of the trees by drought or low temperatures during November is highly important in determining their cold-hardiness.

The data presented by Cooper and Peynado (1959) in table 148 show the mean minimum and maximum temperatures from November through March, for a 10-year period, at various locations in California, Florida, and Texas. To supplement these data, figures were also reported for the number of days from November 1 through March 31 in which the daily air temperatures dropped below 50° F. (10° C.) during cold and warm winters at these same locations (table 149). The authors reported that 50 degrees was the minimum temperature for the active growth of grapefruit trees. Since the trees become dormant in October and November in response to certain internal factors, they usually remain dormant all winter if minimum tempera-

TABLE 148

MEAN MONTHLY MINIMUM AND MAXIMUM AIR TEMPERATURES FOR NOVEMBER THROUGH MARCH FOR THE 10-YEAR PERIOD 1948–57 AT SELECTED LOCATIONS IN CALIFORNIA, FLORIDA, AND TEXAS.*

Locations and sources	November	December	January	February	March	Means
Minimum temperatures (F. and C.)						
Indio, California (United States Date Field Station)	45 (7.2)	38 (3.3)	37 (2.8)	41 (5.0)	47 (8.3)	42 (5.5)
Riverside, California (University of California Citrus Experiment Station)	43 (6.0)	39 (4.0)	37 (2.8)	39 (4.0)	40 (4.4)	40 (4.4)
Orlando, Florida (United States Subtropical Experimental Farm)	54 (12.0)	50 (10.0)	49 (9.4)	52 (11.0)	55 (13.0)	52 (11.0)
Ocala, Florida (United States Weather Bureau Climatological Station)	48 (9.0)	45 (7.2)	45 (7.2)	48 (9.0)	50 (10.0)	47 (8.3)
Weslaco, Texas (Texas Agricultural Experiment Station, Substation No. 15)	56 (13.3)	53 (11.7)	53 (11.7)	56 (13.3)	60 (16.0)	56 (13.3)
Crystal City, Texas (Texas Agricultural Experiment Station, Substation No. 19)	49 (9.4)	44 (6.7)	44 (6.7)	44 (6.7)	54 (12.0)	47 (8.3)
Maximum temperatures (F. and C.)						
Indio, California	82 (28.0)	73 (22.8)	70 (21.0)	76 (24.4)	79 (26.0)	76 (24.4)
Riverside, California	76 (24.4)	68 (20.0)	63 (17.2)	67 (19.4)	69 (20.6)	69 (20.6)
Orlando, Florida	77 (25.0)	73 (22.8)	73 (22.8)	76 (24.4)	79 (26.0)	76 (24.4)
Ocala, Florida	77 (25.0)	72 (22.2)	72 (22.2)	79 (26.0)	80 (27.0)	76 (24.4)
Weslaco, Texas	77 (25.0)	74 (23.3)	74 (23.3)	77 (25.0)	81 (27.2)	77 (25.0)
Crystal City, Texas	74 (23.3)	70 (21.0)	69 (20.6)	74 (23.3)	80 (27.0)	73 (22.8)

*From Cooper and Peynado (1959).

tures lower than 50° F. prevail. The mean minimum winter temperatures at Riverside and Indio, California, were approximately 10 degrees below this minimum temperature essential for growth. The mean minimum temperatures for Ocala, Florida, and Crystal City, Texas, were slightly below this point. Weslaco, Texas, had a mean minimum of 56° F. (13.3° C.), which was the highest value reported for any location. The mean maximum temperatures at all locations were not significantly different, with the exception of those reported for Riverside in December through March, which resulted in a reduction of the over-all mean to 69° F. (20.6° C.).

In addition to the occurrence of minimum air temperatures necessary to induce dormancy, the frequency with which temperatures below 50 degrees occurred was highly related to dormancy and freeze injury (table 149). In cold winters, the air temperatures at Riverside dropped below that reading for 151 days, which amounted to the total number of days in the experimental period. In both cold and warm winters, the temperatures at Riverside and Indio dropped below 50 degrees more frequently than in the other areas. Crystal City was also characterized by generally cooler night temperatures than either Weslaco or Orlando. The low night temperatures at Riverside, Indio, and Crystal City had a tendency to induce greater dormancy and

TABLE 149

NUMBER OF DAYS FROM NOVEMBER 1 THROUGH MARCH 31 IN WHICH DAILY AIR TEMPERATURES DROPPED BELOW 50° F. (10° C.) DURING COLD AND WARM WINTERS AT SELECTED LOCATIONS IN CALIFORNIA, FLORIDA, AND TEXAS.*

Locations and seasons†	Nov.	Dec.	Jan.	Feb.	Mar.	Totals
Cold winters (number of days below 50° F. or 10° C.)						
Indio, California, 1948–49	29	31	31	25	18	134
Riverside, California, 1948–49	30	31	31	28	31	151
Orlando, Florida, 1957–58	4	16	25	22	9	76
Ocala, Florida, 1957–58	11	22	27	22	16	98
Weslaco, Texas, 1950–51	9	15	15	10	7	56
Crystal City, Texas, 1950–51	17	28	25	18	11	99
Warm winters (number of days below 50° F. or 10° C.)						
Indio, California, 1957–58	26	30	29	15	19	119
Riverside, California, 1957–58	27	26	28	24	28	133
Orlando, Florida, 1948–49	1	5	6	2	13	27
Ocala, Florida, 1948–49	2	11	9	..‡	..	..
Weslaco, Texas, 1949–50	7	4	5	6	4	26
Crystal City, Texas, 1949–50	24	12	10	11	12	69

*From Cooper and Peynado (1959).
†Sources are stated in table 148.
‡Blanks mean "data not available."

maintain it for a longer period than was the case at Weslaco and Orlando. The more dormant trees at Riverside and Crystal City showed considerable resistance to freezing temperatures and sustained less injury.

No attempt is being made here to discuss all the many climatic, soil, and cultural factors that contribute to the freeze damage of citrus trees.

Factors That Affect Juice Volume

THE VOLUME as well as the composition of the juice and segments is important from the consumer's point of view. It is of interest that since 1934–35, more juice has been canned in the United States than segments. With the continued utilization of citrus fruits in processed forms, both volume and quality of juice in the fruit have become extremely important to the processor. The following reports are given to show variations in the volume of juice per fruit or per sample of fruit, and some of the factors that may have been responsible for them.

Baier and Higby (1931*a*, *b*) found that juice should be extracted from all portions of the pulp in order to determine its whole volume and composition.

Longfield-Smith (1935*c*) obtained 10 per cent more juice from peeled than from nonpeeled fruit. Different methods of squeezing gave different amounts of juice. The effects of each of these factors are shown by the following figures, adapted from his tables 1 and 2. Each value represents the average of 9 to 13 determinations, and shows the percentages obtained from whole fruits.

Nonpeeled fruit, press squeeze	43.9
Nonpeeled fruit, hand squeeze	37.0
Peeled fruit, press squeeze	47.9
Peeled fruit, hand squeeze	43.6

Longfield-Smith also found that the first squeezing gave higher acids and less soluble solids, and therefore a lower ratio. This indicated that complete squeezing must be done if satisfactory results are to be obtained.

Lueck and Pilcher (1941) and Shearon and Burdick (1948) described several different methods of juice extraction, and gave the necessary precautions to be observed when using each method.

Age of Fruit. Harding and Fisher (1945), in Florida, found that the volume of juice usually increased until the fruit ripened, and then remained rather constant. In Texas, Krezdorn and Cain (1952) reported that juice (by weight) increased as the season advanced, but leveled off in November and December, and that variety appeared to have no effect on the juice volume. In Arizona, Hilgeman (1941) stated that juice volume was not closely correlated with maturity. Croucher (1935), in Jamaica, reported that juice volume, as observed in that area, rapidly increased and reached a maximum considerably before the fruit reached maturity.

Pruning. In studying the influence of pruning on the size and quality of Florida grapefruit, Kretchman and Jutras (1962) noted that total yields were not increased significantly by pruning, but that the pack-out (highly associated with size and grade) was significantly increased. Hedging on 2 or 4 sides plus topping at 12 or 15 feet (about 4 or 5 meters) generally produced the greatest increase in pack-out. The juice content tended to increase with the severity of pruning; the soluble solids and acids decreased, but the ratio of soluble solids to acids showed no significant change.

Locality. The quantities of juice in fruits from different areas, states, and countries are given in this section.

López-Domínguez (1919, 1921) and López-Domínguez and Cady (1921) made a large number of determinations on the changes that occurred in grapefruit during the process of maturation in Puerto Rico. One of these was to determine the changes in juice content of the fruit as affected by variety and stage of maturity. They worked with the Duncan, Marsh, and Triumph varieties. The values presented here are averages from several groves for the seasons 1916–17 and 1917–18. The dates of testing were usually from October to January or September to February; tests were made at biweekly intervals. The juice contents are stated as percentages.

	1916–17	*1917–18*
Duncan	43.47	38.97
Marsh	44.94	41.16
Triumph	33.09	—

These workers found that: (1) the percentage of juice in the fruit increased as the season advanced; (2) the percentage of juice was highest in fruit grown on clay soil; and (3) excessive rainfall caused delay in maturation and increase in juice, but loss in percentage of soluble solids and acids.

Hardy and Rodríguez (1935), in Trinidad, tested the quantities of juice in grapefruit from El Quemado estate, where the soil was rich in lime, and from the Maqueripe estate, where it was highly acid. They used fruit of several grades, but reported values only for choice fruits and culls. They found that the percentage of juice and pulp combined was 76.8 for choice and 75.9 for cull fruit in El Quemado, and 79.2 for choice and 74.2 for culls in Maqueripe. They thus concluded that the choice fruits had a slightly higher proportion of juice and pulp than the culls. For other values from these samples, see table 58 of chapter 2.

Van der Plank and Rattray (1938*a*), in South Africa, determined the effects of storage at different temperatures on the amounts of juice that could be extracted from grapefruit after remaining in storage for 31 days. The temperatures used ranged from 40 to 70° F. (4.4 to 21° C.), at intervals of 10 degrees Fahrenheit. The greatest gain in amount of extractable juice was in fruit stored at 60° F. (16° C.) in boxes with liners; the amount increased from

42.90 to 45.76 per cent, a gain of 2.86 per cent. In other words, under these conditions there would be nearly as much juice in 6 stored boxes as in 7 non-stored boxes. The amount of extractable juice increased at the expense of peel and rag.

Joachim and Panditteseker (1939) concluded, from the results of 62 tests of local and imported fruit, that Ceylon grapefruit was similar in quality to that of other countries. They tested and compared 17 local samples and 7 samples imported from South Australia, California, South Africa, and Rhodesia (mainly Marsh). The juice content of the Ceylon samples ranged from 30.5 to 55.2 per cent.

Ellis and Tai (1949) determined the volume of juice in fruit from different parishes in Jamaica. Sometimes the samples came from only 2 or 3 groves per parish, and sometimes from as many as 28; 90 different groves were tested. Only mature fruit was used. The values given in table 150 include the averages from each parish, and the volume of juice is expressed as percentage by weight. From the results, they concluded that grove variation within a given district was greater than the variations between different districts.

Table 151 from Harding and Fisher (1945) gives a general picture of the juice content of Marsh and Duncan grapefruit grown in several areas in Florida. Both varieties were on rough-lemon rootstock. The determinations were made at different times of the year, as shown, from 1939–40 to 1942–43. The authors reported that the Marsh fruits consistently contained more juice than the Duncan, probably owing to the lack of seeds in Marsh. Similar tables

TABLE 150

THE AVERAGE PERCENTAGES OF JUICE IN GRAPEFRUIT SAMPLES FROM DIFFERENT PARISHES IN JAMAICA.*

Parish designations	Number of groves tested	Juice (per cent by weight)		
		Maximum	Minimum	Averages
A	6	55.3	43.0	47.8
B	9	63.7	33.8	44.1
C	3	47.1	38.6	46.4
D	5	51.6	41.5	47.2
E	2	39.9	34.5	37.2
F	4	46.7	38.8	42.6
G	28	68.8	30.8	40.0
H	23	42.8	32.7	38.4
I	10	53.1	32.7	44.2
	90			
Mean juice values		52.1	36.4	43.1

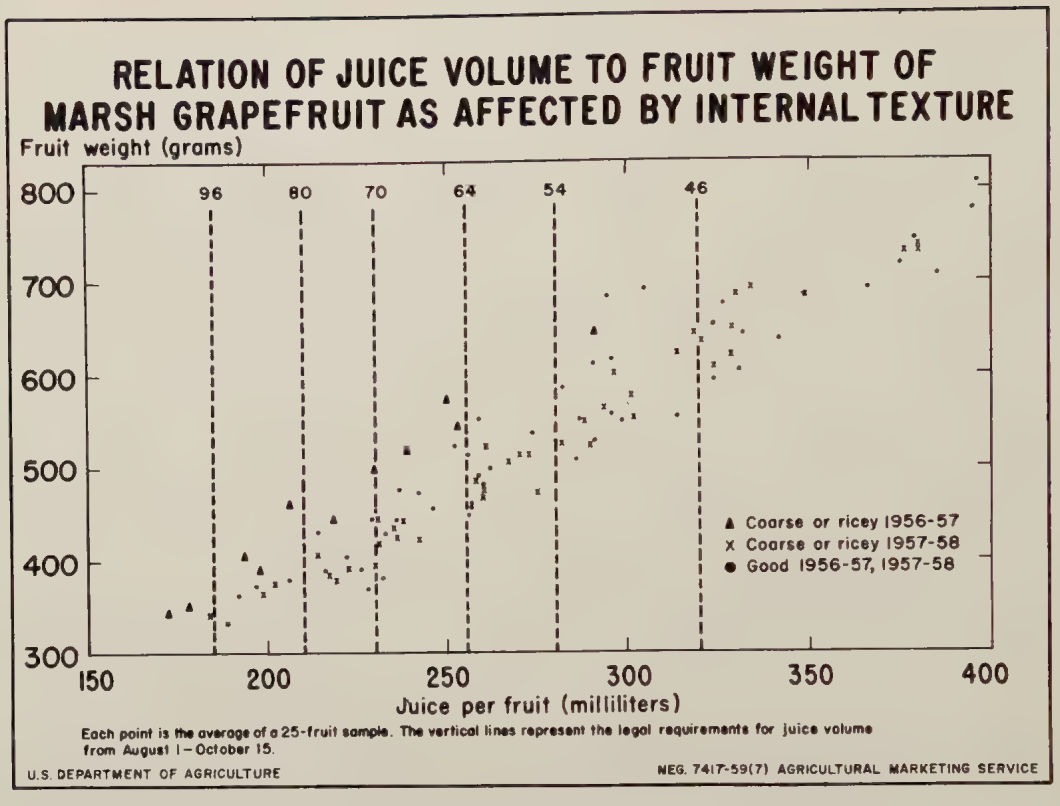
*Adapted from Ellis and Tai (1949).

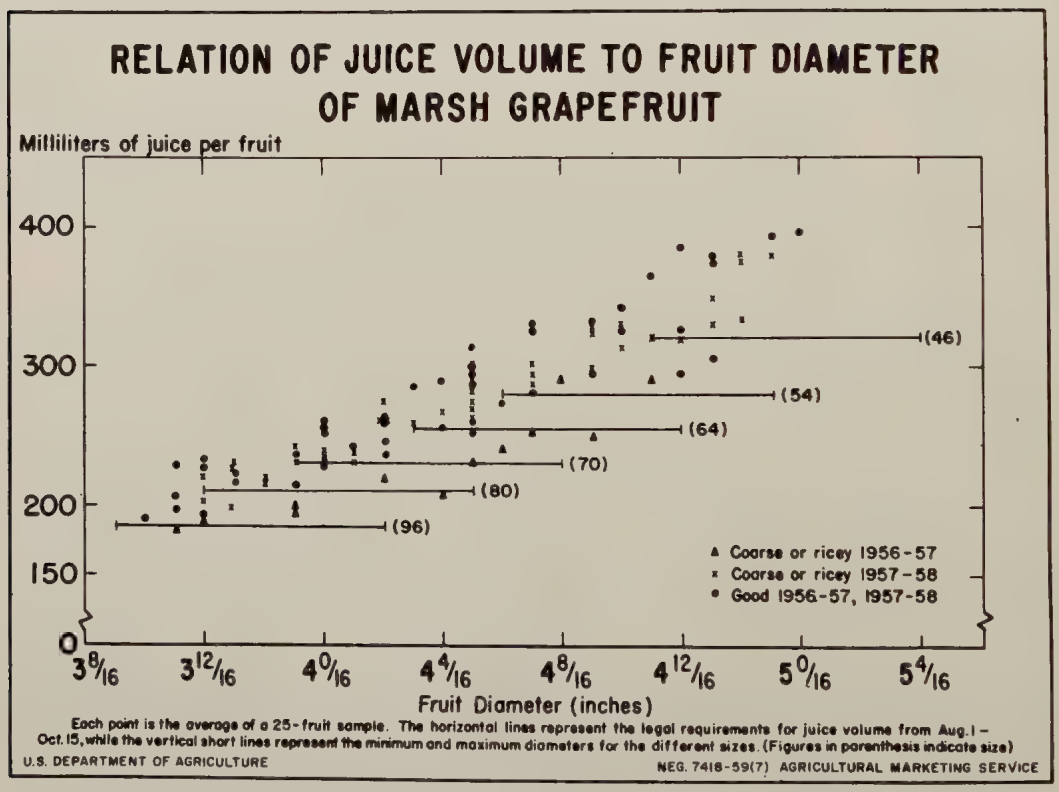
TABLE 151

JUICE CONTENT OF MARSH AND DUNCAN GRAPEFRUIT GROWN ON ROUGH-LEMON ROOTSTOCK IN DIFFERENT AREAS OF FLORIDA.*
(Milliliters of juice per 100 grams of fruit.)

Picking periods	Davenport		Lake Hamilton		Windermere		Bradenton		Fort Pierce		Homestead	
	Marsh	Duncan	Marsh	Duncan	Marsh	Duncan	Marsh	Duncan	Marsh	Duncan	Marsh	Duncan
August 25–September 2	33	30	30	28	36	..	34	..	31	29	38	38
September 25–30	41	36	37	36	45	..	44	..	39	37	48	43
October 23–28	45	42	43	41	49	..	48	..	46	42	51	45
November 20–25	46	44	42	40	49	..	50	..	49	46	51	47
December 18–23	46	45	44	43	52	..	53	..	51	48	54	50
January 15–20	47	45	44	44	50	..	52	..	50	47	52	48
February 12–23	49	43	45	43	49	..	53	..	51	46	51	49
March 11–16	47	45	45	44	50	..	53	..	52	48	47	48
April 8–13	48	44	46	45	48	..	53	..	51	48	48	51
May 6–11	49	45	46	46	47	..	54	..	52	48	49	50

*Adapted from Harding and Fisher (1945), various appendix tables.




RELATION OF JUICE VOLUME TO FRUIT DIAMETER OF MARSH GRAPEFRUIT
Milliliters of juice per fruit
400
300
200
150
0
(46)
(54)
(64)
(70)
(80)
(96)
▲ Coarse or ricey 1956-57
x Coarse or ricey 1957-58
● Good 1956-57, 1957-58
3 8/16
3 12/16
4 0/16
4 4/16
4 8/16
4 12/16
5 0/16
5 4/16
Fruit Diameter (inches)
Each point is the average of a 25-fruit sample. The horizontal lines represent the legal requirements for juice volume from Aug. 1 – Oct. 15, while the vertical short lines represent the minimum and maximum diameters for the different sizes. (Figures in parenthesis indicate size)
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Figs. 97 (top) and 98 (bottom). (From Long *et al.*, 1959.)

for the juice content of these 2 varieties when grown on sour orange are also given in the appendix to their paper. The conclusion was drawn that the rootstock did not influence the amount of juice to any appreciable extent.

Long *et al.* (1959) reported that although large variations occurred in any physical or chemical characteristic of grapefruit, high correlations actually existed between fruit weight, fruit diameter, juice volume, and internal texture. This is substantiated by the data presented in figures 97 and 98, which show the relationships between juice volume and fruit weight as well as between juice volume and fruit diameter. The more closely these points fall along a given line, the higher is the correlation coefficient. The data indicate that good-textured fruits of the same diameter usually yield more juice and

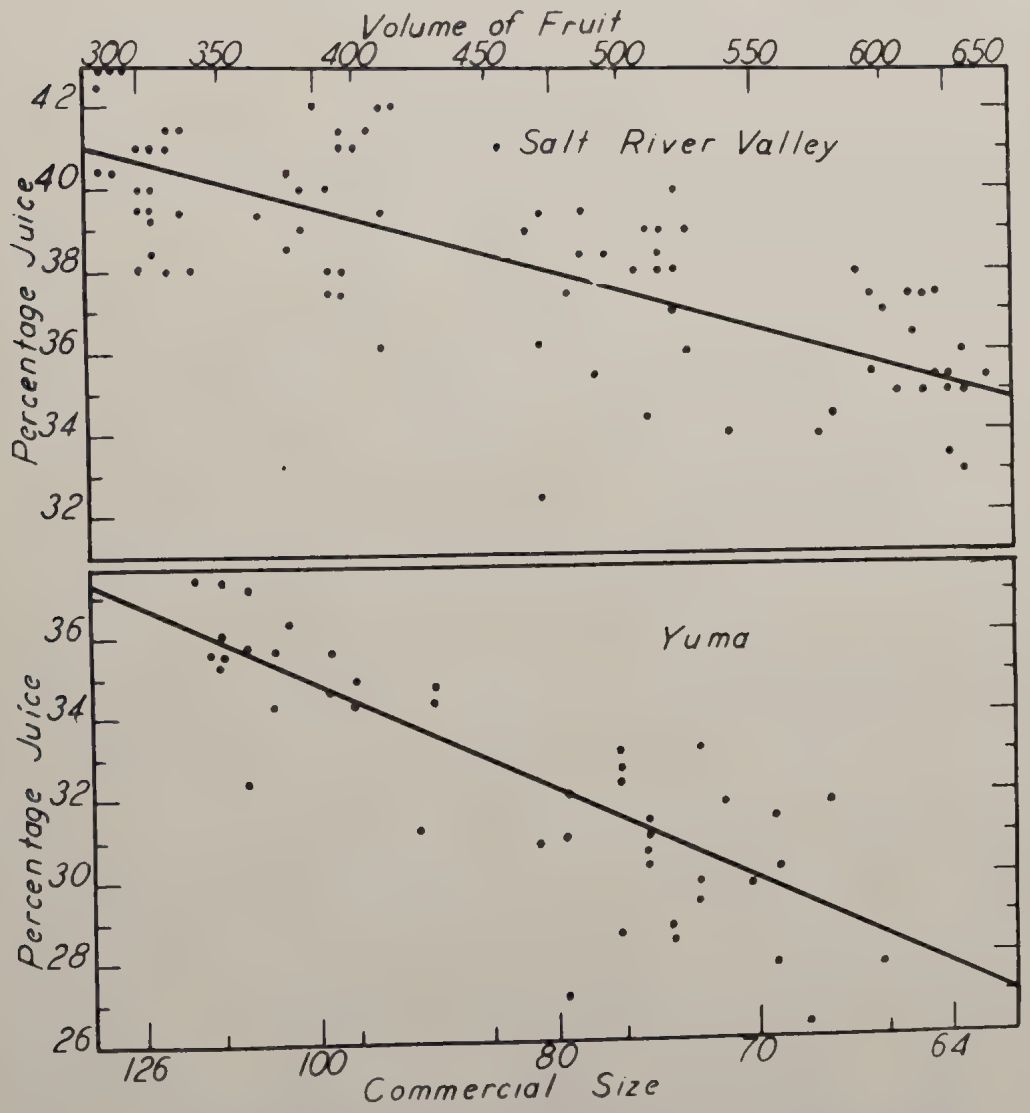


Fig. 99. Scatter diagrams showing the linear relationships between size and percentage of juice in citrus fruits from packing houses in the Salt River Valley and Yuma, Arizona. (From Hilgeman, 1941.)

are heavier than coarse- or ricey-textured ones. The horizontal lines designate the legal requirements for juice volume, and the short vertical lines show the variation and overlapping of the diameters of the different fruit sizes. In general, an increase in juice volume (milliliters per fruit) is positively correlated with an increase in fruit weight, and also with an increase in fruit diameter. The weight of individual grapefruit of a given size is highly variable, as shown by the fact that size 96 fruit harvested in the fall weighed from

TABLE 152

AVERAGE SEASONAL CHANGES IN PERCENTAGES OF JUICE BY WEIGHT IN WHOLE FRUIT IN 2 AREAS OF ARIZONA.*

Date	Salt River Valley (12 groves)					Yuma (5 groves)				
	1937	1938	1939			1937	1938	1939		
			Averages	High†	Low†			Averages	High	Low
September 8..	....	37.1	32.0	36.2	26.3	31.2	36.3	35.5	38.8	32.7
16..	39.7	37.0	35.1	39.0	31.4	41.6	39.8	37.8	42.0	34.6
23..	41.7	40.2	35.8	40.7	30.6	42.6	41.6	39.0	41.1	36.9
30..	43.7	42.3	38.5	43.9	34.0	42.2	43.4	41.9	46.1	39.3
October 7.....	43.8	43.0	41.8	45.2	37.1	42.5	44.2	43.5	47.8	41.7
14.....	45.8	46.5	43.2	47.3	38.7	45.7	45.9	44.8	47.6	42.7
21.....	46.3	45.8	45.3	49.0	41.8	45.9	47.2	46.4	51.1	44.1
28.....	47.3	47.9	46.5	49.9	43.3	47.3	47.2	46.9	49.8	44.3
November 3..	48.1	46.6	46.2	50.7	42.2	48.4	48.4	47.4	50.9	45.0
11..	47.7	48.5	47.8	51.8	43.5	48.1	48.3	48.7	53.0	45.5
18..	48.0	49.5	49.6	53.8	46.4	49.0	49.0	48.8	51.6	46.6
25..	48.5	49.8	49.9	54.7	45.9	49.1	48.4	48.0	50.3	46.5
December 2...	49.8	49.1	49.6	57.4	44.1	49.7	50.6	48.8	54.0	47.1
9...	49.3	51.8	50.7	54.1	48.6	50.2	49.6	48.9	53.2	46.4
16...	47.2	51.4	50.5	53.0	48.2	49.1	48.7	47.1	49.1	45.3
23...	46.0	48.6	49.6	54.1	46.5	47.7	47.8	49.4	52.5	47.8
30...	47.8	49.9	49.0	53.5	46.3	....	....	48.3	51.6	47.1
January 6.....	48.3	49.5	50.3	54.5	46.9	48.9	48.5	49.3	52.9	47.1
13.....	....	50.4	50.1	54.1	47.4	48.2	48.3	48.2	52.6	45.5
20.....	....	50.6	49.8	54.4	44.4	....	47.2	48.1	50.9	45.9
28.....	....	50.2	....	....	....	....	....	....	....	....
February 21...	....	50.4	48.1	51.9	43.8	....	....	....	....	....
March 21.....	....	47.7	45.9	51.2	39.5	....	....	....	....	....
April 20.....	....	49.2	45.5	50.0	40.3	....	....	....	....	....
May 22.....	....	50.7	47.9	52.4	43.3	....	....	....	....	....

*From Hilgeman (1941).
†Highest and lowest values found on the days when tested.

288 to 451 grams, with a mean of 365 grams, and similar fruit harvested in the spring varied from 307 to 491 grams, with a mean of 385 grams.

Hilgeman (1941), working with Arizona grapefruit, made a study of different methods of determining the juice content of citrus fruits. He stated that percentages based on the whole fruit were affected by the weight of the peel, and were not entirely satisfactory. In this method, corrections would have to be made for fruit size. The data in figure 99 show the effects of volume and

TABLE 153

AVERAGE SEASONAL CHANGES IN PERCENTAGES OF JUICE BY WEIGHT IN PEELED FRUIT IN 2 AREAS OF ARIZONA.*

Date	Salt River Valley (12 groves)					Yuma (5 groves)				
	1937	1938	1939			1937	1938	1939		
			Averages	High†	Low†			Averages	High	Low
September 8..	....	61.4	59.8	65.5	49.0	....	63.4	64.3	65.9	59.2
16..	66.0	61.0	57.6	68.5	55.5	69.2	67.2	67.3	70.8	63.2
23..	68.5	64.4	59.9	65.3	56.4	68.7	68.3	66.5	70.6	59.0
30..	69.6	66.2	65.7	71.5	58.6	66.5	69.3	70.3	72.9	70.2
October 7.....	69.8	67.5	69.2	73.5	63.2	69.2	68.5	71.2	73.3	69.5
14.....	72.0	72.1	69.0	72.8	60.5	70.8	69.4	72.2	74.2	70.0
21.....	72.2	69.5	71.6	75.5	65.9	72.8	72.8	73.6	76.8	71.2
28.....	73.7	73.4	72.8	76.4	66.8	73.7	72.9	73.6	75.7	70.8
November 3..	74.8	71.4	74.1	77.0	71.1	75.8	73.5	74.2	76.9	72.6
11..	73.6	74.4	74.4	78.8	67.1	74.6	73.7	75.2	78.3	71.3
18..	75.1	75.4	75.9	79.2	71.7	75.5	74.7	75.5	77.4	73.6
25..	75.7	75.6	75.4	78.8	67.6	76.1	76.0	74.8	76.8	72.3
December 2...	78.0	73.8	75.9	80.4	68.6	76.1	74.5	76.0	78.6	74.4
9...	77.6	78.6	77.1	79.7	70.6	78.0	77.8	76.0	80.0	74.0
16...	75.6	78.2	77.4	80.6	74.5	75.5	77.1	74.3	75.8	73.1
23...	73.0	75.4	78.6	81.0	75.9	74.3	77.1	78.2	79.5	77.4
30...	76.2	77.7	76.8	79.7	73.7	....	74.4	76.5	78.8	74.2
January 6.....	78.2	77.1	77.5	82.5	75.4	77.2	77.3	77.5	79.0	76.3
13.....	....	78.6	78.2	81.7	77.0	76.1	77.9	77.9	81.3	76.1
20.....	....	78.2	78.6	82.4	72.0	....	77.7	76.6	78.5	73.4
28.....	....	77.3	....	....	....	....	....	....	....	....
February 21....	....	77.6	76.9	79.8	74.8	....	....	....	....	....
March 21.....	....	76.7	77.7	81.4	73.8	....	....	....	....	....
April 20.....	....	79.0	76.0	79.5	71.6	....	....	....	....	....
May 22.....	....	78.8	76.3	78.9	73.6	....	....	....	....	....

*From Hilgeman (1941).
†Highest and lowest values found on the days when tested.

commercial size of the fruit on the yield of juice obtained. In other words, the smaller the volume of fruit and the greater the number per commercial box, the greater is the percentage yield of juice. (*See page 359.*)

The peel affects the percentages by volume even more than the percentages by weight because the specific gravity of the peel is less than that of the edible portion. Thus, a greater portion of the fruit is peel when determined on a volume basis. The third and most accurate method is to calculate the percentage of juice, based on peeled fruit. As determined by Hilgeman (1941), table 152 gives the percentages of juice by weight in whole fruit, and table 153 shows the percentages of juice by weight in peeled fruit. The 2 methods will be seen to have a marked contrast.

The percentages of juice in Texas grapefruit as affected by irrigation, age of tree, and locality where grown are shown by table 154, from Wood and Reed (1938). The fruit from heavily irrigated trees contained an average of 0.73 per cent less juice than that from trees that had been normally irrigated. On the other hand, fruit from trees that had been lightly irrigated contained 1.26 per cent more juice than that from normally irrigated trees. The fruit from 10-year-old trees contained 2.71 per cent more juice than that from 15-year-old trees. The fruit from La Feria plot contained relatively high amounts of juice because the trees had not been irrigated, and that from the Mission plot had the least because the trees bloomed 2 months later than those in the other plots.

Fruit picked from the Engelman plot had slightly more juice on certain dates than that from La Feria plot. There appeared to be a definite correlation between decrease in rag and increase in juice.

Hilgeman (1941), in evaluating the effects of irrigation on grapefruit maturation, reported that no significant changes in the Brix:acid ratios could be correlated with irrigation treatments, and that irrigation might retard the normal rate of increase in percentage of juice by volume. To explain these unproven phenomena, it was suggested that under conditions of low soil moisture, fruits might decrease in diameter, and after applying water to the soil, a rapid increase in fruit volume would occur. If this increase were confined largely to the peel, the percentage of juice, on a volume basis, would probably be reduced. The irrigation data on which the above statements were based were obtained by recording the changes before and after irrigation and comparing them with the normal seasonal trends that were observed for the same period.

In making maturity studies on Marsh grapefruit in California, R. H. Higby (unpublished) determined the average juice contents of fruit from 4 different parts of the state at different times of the year. The dates of testing, the average weights, and the percentages of juice per fruit from the various localities are given in table 155, which also states the source reference.

TABLE 154

PERCENTAGES OF JUICE (BY WEIGHT) IN TEXAS GRAPEFRUIT, AS AFFECTED BY IRRIGATION, AGE OF TREES, AREA WHERE GROWN, AND DATES OF PICKING (1934–35).*

Cultural conditions	Oct. 22	Nov. 6	Nov. 19	Dec. 4	Dec. 21	Jan. 7	Jan. 29	Averages
Normally irrigated	42.40	45.62	46.63	47.23	44.85	46.85	41.92	46.30
Heavily irrigated	40.27	44.34	45.39	47.81	43.73	46.96	49.63	45.57
Lightly irrigated	43.03	50.24	46.14	49.02	46.14	47.75	50.60	47.56
Trees 10 years old	44.94	48.75	48.13	48.78	49.46	51.52	50.32	49.01
Trees 15 years old	42.40	45.62	46.63	47.23	44.85	46.85	41.92	46.30
From La Feria plot†	42.58	42.37	43.01	46.26	47.90	50.15	50.09	46.19
From Mission plot	33.53	31.32	32.68	37.88	39.23	42.62	43.64	37.58
From Engelman plot	42.76	42.42	43.23	47.09	48.09	49.00	51.23	46.53

*Adapted from Wood and Reed (1938), various tables.

†The dates of picking from these 3 plots were not exactly the same as for those given above. The season was 1935–36, but the picking dates also ranged from October to January, with intervals similar to those of the 1934–35 pickings.

TABLE 155

PERCENTAGES OF JUICE IN MARSH GRAPEFRUIT GROWN IN DIFFERENT AREAS OF CALIFORNIA.*

Dates (1931–32)	Fruit weight (grams)	Juice (per cent by weight)
Average values for 8 localities in the Imperial Valley		
September 23	224	34.5
October 15	247	40.9
October 30	289	42.4
November 15	323	45.4
December 1	301	46.4
Average values for 5 localities in the Coachella Valley		
September 22	292	36.2
October 15	301	43.1
November 1	354	46.3
November 15	353	47.3
December 2	375	47.9
Average values for 17 localities in southern California		
February 16	267	41.6
March 2	257	42.6
March 16	277	42.3
March 30	277	42.9
April 15	281	43.4
May 1	289	42.7
May 18	300	44.8
June 1	300	44.8
Average values for 10 localities in central California		
October 13	222	34.6
November 3	231	37.6
November 17	258	39.9
December 3	256	39.6
December 16	259	39.9
January 1	259	39.8

*Adapted from R. H. Higby: Maturity work on citrus fruits. Sunkist Growers Research Department, File 15, Vol. 7:1–96. Unpublished data gathered in 1931–32.

Stewart and Parker (1954), in their work on the effects of growth hormones on Marsh grapefruit grown in different areas in southern California, found the following percentages of juice (by weight) in 4 localities.

University of California Citrus Experiment Station, Riverside	45.3
Prenda	44.3
Saticoy	39.5
Sespe	44.6 and 45.9

Further data on the relative amounts of juice in Marsh grapefruit grown in California are shown in table 156, taken from Rygg and Getty (1955). The values are from fruit from 2 desert communities in southern California—El Centro and Brawley. In these areas of Imperial County, the fruit reaches maturity in November and December.

Factors That Affect Flavor

PUBLIC DEMAND for an edible product depends largely on its flavor: taste plus aroma. This definitely applies to grapefruit, and is influenced by the time of harvest and by premarket treatments of the fresh fruit, such as washing, drying, polishing, sterilizing, waxing, grading, sizing, coloring or degreening, packing, and storage and transportation temperatures. The flavor or taste of the juice and segments is influenced by the manner in which the processing is done and the treatment of the processed products. The organoleptic properties, as demonstrated by taste-panel procedures, have been shown to be highly correlated with the edible qualities of the fresh and processed products. Taste and flavor are directly related to the constituents that compose the product, and in some instances, taste qualities have been correlated with chemical structure.

No attempt will be made here to go into the chemistry of flavor. Hornstein and Teranishi (1967) gave an extended discussion of the fundamental principles related to taste and odor and the analytical procedures used to elucidate the problem of flavor. The following text deals with some of the factors and conditions that affect the flavor of mature grapefruit and of its fresh and processed juice.

In Fresh Juice and Fruit. Baier (1936), in California, pointed out that the juice of 2 different fruits may have the same soluble solids:acids ratio and at the same time be very different in palatability and even in sweetness. This is because of differences in ash and other buffers (substances that affect the active but not the titratable acidity), flavoring constituents, essential oils, and bitter substances.

Sites (1944) found that at the end of a 5-year differential fertilizer treatment, in which 5 plots received nitrogen, phosphorus, and potassium, and

TABLE 156

SEASONAL VARIATIONS IN AMOUNTS OF JUICE AND CHEMICAL CONSTITUENTS IN MARSH GRAPEFRUIT FROM 2 DESERT AREAS OF CALIFORNIA.*

Treatment and picking dates	Weight per fruit (grams)	Juice content of whole fruit (per cent)	Total soluble solids (per cent)	Total acid (per cent)	Solids: acid ratios
FRUIT FROM EL CENTRO					
1945–46:					
October 24	281	47	12.10	1.70	7.12
December 5	335	47	12.32	1.67	7.38
January 9	348	50	12.67	1.67	7.59
February 13	409	45	12.49	1.68	7.43
March 20	491	43	12.18	1.49	8.17
April 24	479	46	11.74	1.32	8.89
June 5	443	49	11.03	1.09	10.12
1946–47:					
October 2	209	43	11.97	1.97	6.08
November 7	296	45	12.14	1.92	6.32
December 11	292	45	12.86	1.88	6.84
January 23	345	47	13.29	1.93	6.89
March 5	376	45	12.48	1.80	6.93
April 15	418	47	11.78	1.55	7.60
May 28	434	51	11.54	1.30	8.88
July 16	352	46	12.37	1.03	12.01
1947–48:					
September 10	182	37	11.84	2.26	5.24
October 1	220	43	12.31	1.89	6.51
October 29	337	49	11.73	1.76	6.63
December 3	279	52	12.78	1.84	6.95
January 28	354	48	13.41	2.04	6.57
March 17	388	59	12.71	1.78	7.14
May 12	403	57	11.69	1.49	7.85
July 8	361	56	11.52	1.05	10.97
FRUIT FROM BRAWLEY					
1945–46:					
October 25	312	39	12.05	1.78	6.77
December 5	359	44	12.13	1.72	7.05
January 10	364	42	12.10	1.73	6.99
February 14	441	39	12.54	1.70	7.38
March 21	399	44	11.95	1.63	7.33
April 25	406	44	11.99	1.51	7.94
June 5	386	47	11.31	1.34	8.45
July 18	366	47	12.51	0.97	12.88

*From Rygg and Getty (1955).

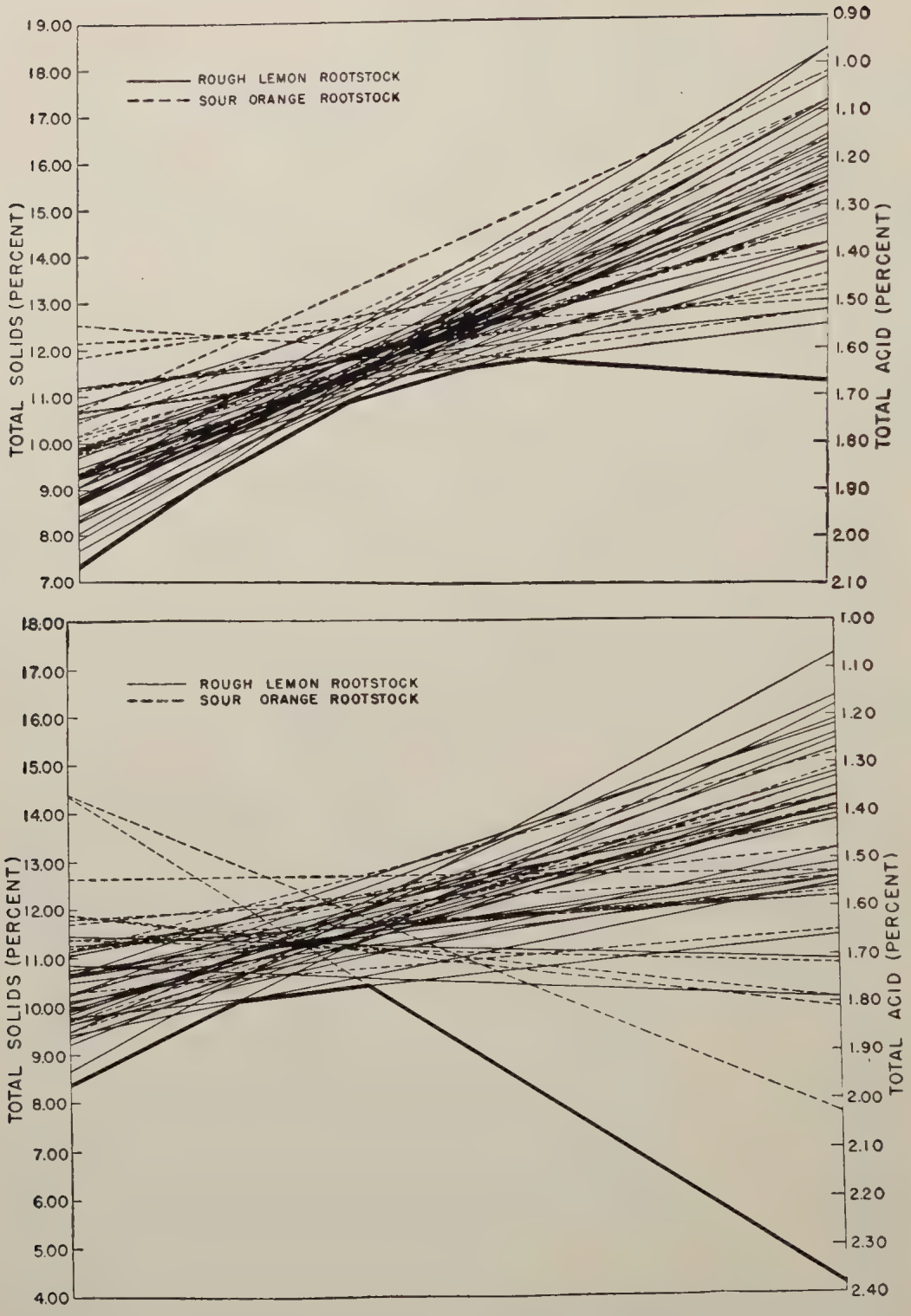
5 other plots received these plus magnesium, zinc, and copper, the latter produced grapefruit with a sweeter taste, a generally improved flavor, and showed an increase in yield. This was true for both the Marsh and Duncan varieties. (*See table 134 on page 311, giving data for Duncan.*)

Sinclair and Bartholomew (1947) called attention to the fact that soluble constituents of the juice, other than sugars and acids, are important in taste sensations. They stated that about 15 per cent of the soluble constituents of orange juice is composed of substances other than sugars and acids. This fraction consists of inorganic compounds, amino acids, ascorbic acid, and small amounts of pectins, essential oils, esters, and glycosides. Substances in this fraction are relatively unstable chemically, whereas the soluble sugars and acids of citrus juices are stable. The occurrence of off-flavors in either natural or processed orange juice is largely owing to the oxidation and decomposition of substances in this relatively small fraction of the soluble solids.

Harding (1947*b*) reported that a statistical study of taste ratings by a panel of judges showed a definite association between the palatability of citrus fruits and their contents of total soluble solids and total acids. As a rule, the tasters rated a fruit as satisfactory only if it had a higher solids:acids ratio than that needed to pass state maturity laws. Harding (1952) also stated that fruit which had attained maximum weight and volume of juice, a medium-high content of total solids, and a relatively low total acid content was rated the most palatable.

The nomographs of figures 100 and 101 show the relationships of the total solids and total acid contents to consumer approval or disapproval of representative samples of Marsh and Duncan grapefruit on rough-lemon and sour-orange rootstocks (Harding and Fisher, 1945). The consumer evaluation of these samples was based on the results of a taste panel. Marsh grapefruit with a pleasantly tart flavor had total solids of 7.37 to 12.53 per cent and a total acid content of 0.97 to 1.67 per cent (figure 100). Similarly, Duncan grapefruit with a pleasantly tart flavor had a total solids value of 8.37 to 14.39 per cent and total acid value of 1.07 to 2.38 per cent (figure 101). The two nomographs are distinctly different, in that the acceptable Marsh variety contained a lower concentration of both total solids and total acids than the Duncan variety. Such varietal differences in consumer acceptability, as determined by the taste test, strongly indicate that 2 maturity standards, one for Marsh and the other for Duncan, should be used. For related information, see Harding (1952).

Although flavor is a physiological sensation, the taste of citrus fruits and juices is partially related to their contents of soluble solids (sugars as Brix), total acidity, and pH of the juice, and these particular compositional factors are presently used to measure the maturity index for both oranges and grapefruit. It follows that the acid taste of citrus fruits is measured by the organic



Figs. 100 (top) and 101 (bottom). *Top:* Nomograph showing total solids and total acid percentages in Marsh grapefruit when first rated by the taste test to meet consumer approval. Data were obtained in 4 seasons (1939–40 through 1942–43), and included tests on fruit from 2 different rootstocks, and from trees sprayed with lead arsenate. The heavy line indicates minimum total solids and maximum total acid of acceptable fruit. *Bottom:* Same data for Duncan grapefruit. (From Harding and Fisher, 1945.)

acid content of the juice; the sweet taste is measured by the content of soluble sugars. This balance between sweetness and tartness is defined by the legal standards of quality which specify limits of the Brix:acid ratio, a value commonly used in the citrus industry to express the relationship between sweetness and tartness. There is consequently a high degree of correlation between the Brix:acid ratio and the degree of tartness in citrus juice.

Kilburn (1958) demonstrated that tartness is inversely related to the Brix:acid ratio (figure 102). The tart sensation of citrus juices, like other biological reactions, is directly associated with the hydrogen ion concentration; consequently, the sour taste (or tartness) is inversely related to the pH. A highly significant correlation exists between these 2 variables

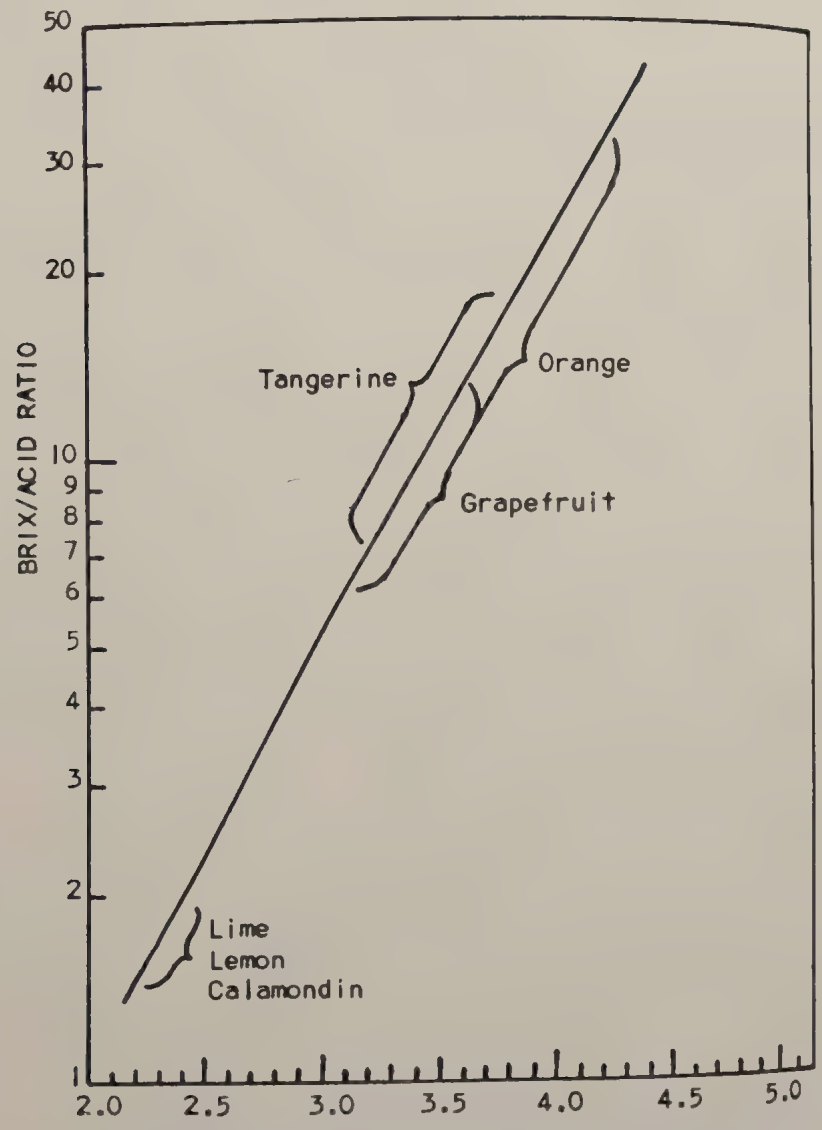


Fig. 102. Regression line showing relationships between Brix:acid ratios and pH. The labeled braces indicate range of variation. (From Kilburn, 1958.)

in citrus juices. As Kilburn noted, "Brix:acid ratio" is a valuable term to those who have had sufficient experience in tasting citrus juices to enable them to relate the taste to the ratio. The regression line showing the relationship between the Brix:acid ratio and pH of citrus juices is illustrated in figure 102. From this correlation, the relative tartness of the juices can be expressed in terms of either Brix:acid ratio or pH. During the seasonal growth and maturation of oranges and grapefruit, the total acid concentration (mostly citric) in the juice decreases, while total soluble solids (Brix) increase with a concomitant increase in pH.

The regression line (figure 103) showing the relationship between the acid:Brix ratios and pH of citrus juices indicates that the pH, within a limited range, is a function of the logarithm of the citric acid concentration if the juice samples are adjusted to a uniform Brix. It follows that the re-

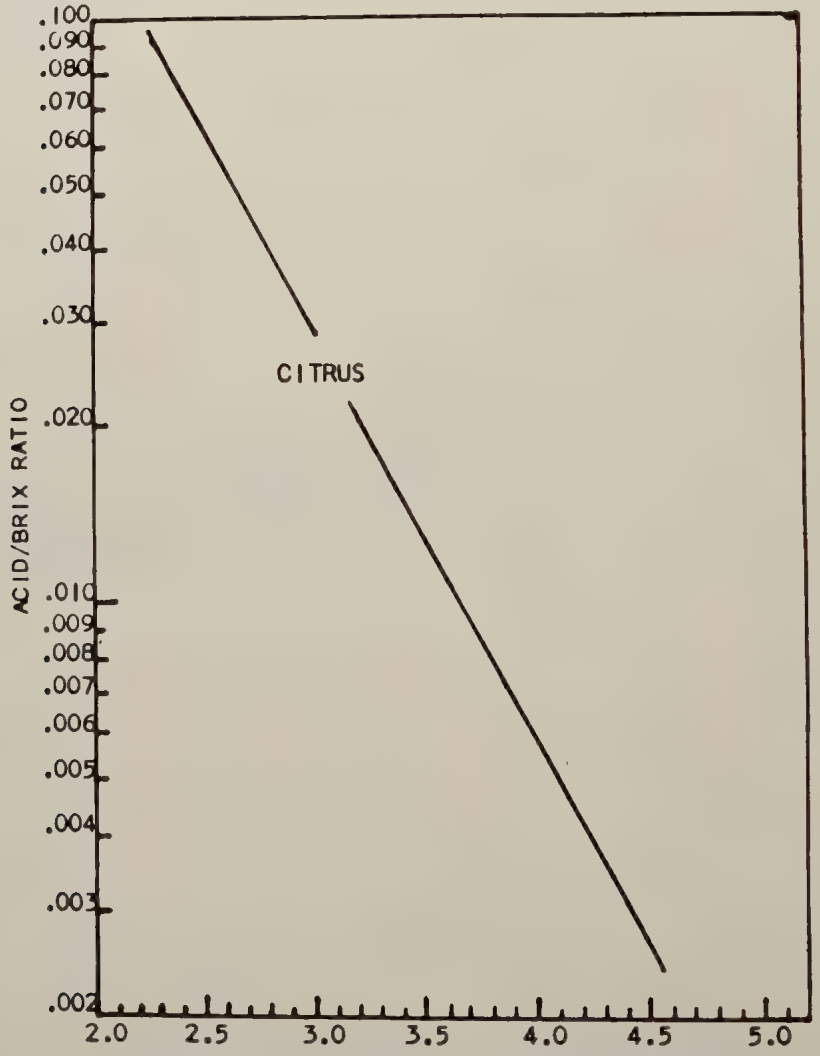


Fig. 103. Regression line showing relationships between acid:Brix ratios and pH. (From Kilburn, 1958.)

lationship between tartness and citric acid concentration is frequently quite variable as a result of changes in the soluble-solids contents or Brix values of the juices.

Kilburn and Davis (1959) reported that grapefruit juices maintained an average salt concentration (calculated as milliequivalents per gram of soluble solids) throughout the entire season, and (as would be expected) that the pH of grapefruit juice varied primarily with the differences in free acidity (figure 104).

In Storage. Van der Plank *et al.* (1938), in Capetown, noted that storage temperatures of 50 to 55° F. (10 to 13° C.), especially early in the season, were best for retaining the flavor and texture of fresh grapefruit. Toward the close of the season, these temperatures might cause staleness of flavor, and lower ones should be used.

The flavor and palatability of grapefruit are highly correlated with the stage of maturity at the time the fruit is harvested. Storage conditions, such as temperature, humidity, modified atmosphere, and others, are designed to maintain the harvested fruit in its fresh condition over an extended period. This subject is discussed in greater detail in chapter 5, entitled "Postharvest Changes in Grapefruit."

In Processed Juice. The major problem in processing citrus fruits is to preserve the natural fresh flavor and to prevent the development of off-flavors. Processing techniques are continually being improved, with the specific purpose of maintaining and preserving the fresh flavor during processing and storage. The flavoring components are present in the fruit in such small amounts that large volumes of fruit are required to produce a few grams of aromatic flavoring substances. In order to eliminate or prevent off-flavors in processed citrus juices, the first requirement is to identify and determine the quantity of the various flavoring constituents in both the fresh and processed products. Consequently, the chemistry of flavor and off-flavor in foods is a highly active area of research which is of extreme importance, not only to the citrus industry but to the food-processing industry as a whole.

Mottern and von Loesecke (1933) were among the first to improve the keeping quality of grapefruit juice by de-aeration and pasteurization. They also found that the juice kept better if limonene was removed from the oil it contained; this did away with the turpentine-like flavor in the aged product.

Veldhuis (1951), in Florida, stated that acids, sugars, and minerals were almost unaffected by processing, but that pasteurization impaired the flavor, and that lower storage temperatures tended to prevent off-flavors. He added that research was making progress in avoiding off-flavors caused by changes in fatty materials, peel oil, and other volatile or nonvolatile compounds.

Moore (1949) made comparative tests on bottled and canned grapefruit juices that had been in storage for 1, 3, 6, 12, and 18 months, as well as non-

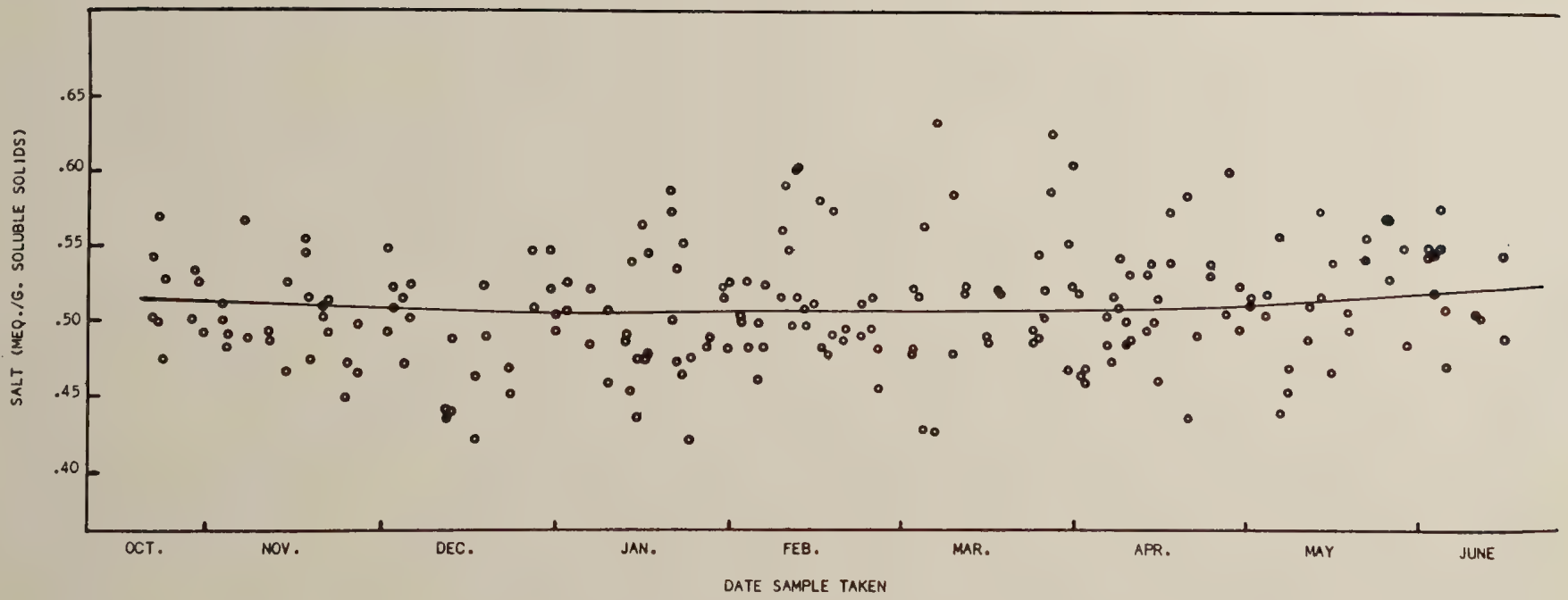


Fig. 104. Seasonal changes in the salt content of grapefruit juice. (From Kilburn and Davis, 1959.)

stored products. He found that the canned juice had the best flavor and appearance on all dates, as well as at the outset.

Marie Jones (1951) stated that it formerly had been difficult to make a good grapefruit concentrate, because the substances imparting its flavor were at least partially destroyed during processing. Investigators have since found that at least most of the flavor could be retained by boiling off the excess water under vacuum at 72° F. (22.2° C.).

Naringin Content. Zoller (1918*a, b*), McNair (1926–27), and Camp *et al.* (1932) were among the earlier workers who determined the effects of naringin on the taste of grapefruit juice. Camp and associates stated that the bitter taste in prepared juice on aging was of glycosidal origin. The tendency toward bitterness was noted to decrease as the fruit became more mature.

Maurer *et al.* (1950) pointed out that the glycoside (naringin) in grapefruit was an important factor in determining its flavor, and that juice containing more than 0.07 per cent naringin possessed an immature flavor. They also noted that the naringin content of the juice offered certain advantages as a measure of maturity over the usual Brix:acid ratio.

Horowitz (1964), in studying the chemical properties of the principal flavanones in citrus, submitted data which showed that taste could be correlated with the chemical structure of the phenolic glycosides. In an earlier paper, Horowitz and Gentili (1961) correlated the bitterness of certain flavanone 7-rhamnoglucosides occurring in citrus with the structure of the disaccharide portion of the molecule. They showed that neohesperidin possessed bitterness, but to a much smaller degree than naringin. Both of these compounds contained the same disaccharide, having the chemical structure of 2-*O*-L-rhamnosyl-D-glucose, in which rhamnose had an alpha configuration. Hesperidin, which is tasteless, and neohesperidin, which is bitter, differ in structure only in the place of attachment of rhamnose to the glucose moiety. Rutinose, the disaccharide present in hesperidin, and neohesperidose, the disaccharide of neohesperidin, are isomeric substances, having the structure of 6-*O*-α-L-rhamnosyl-D-glucose. These scientists discovered several flavanone 7-rutinosides which were tasteless in various citrus species. From their data, it appears that bitterness and nonbitterness in this group of compounds were found to be primarily dependent upon the location at which rhamnose was attached to glucose.

In addition to naringin, Horowitz and Gentili (1961) found, in the peel of Marsh Seedless grapefruit, a flavanone (mp 212–213°) which, on acid or enzymatic hydrolysis, yielded rhamnose, glucose, and (—)-isosakuranetin (4′-methoxy-5,7-dihydroxy-flavanone) (mp 192–194°). The infrared spectra and the melting-point data supported the conclusion that this glycoside from grapefruit was identical with poncirin, a glycoside found in the trifoliate orange (*Poncirus trifoliata*). The ratio of poncirin to naringin was about 1 to

5 in grapefruit samples collected at various time intervals during the growing season.

Horowitz (1961, 1964) presented data on the relative taste and bitterness of phenolic rhamnoglucosides, phenolic glucosides, and aglycones, using quinine dihydrochloride as a standard of reference for bitterness (tables 157 and 158). The flavanone aglycones tested were free of bitterness (table 157). Naringin and poncirin had the highest potencies for bitterness of any flavanone glycosides thus far observed, and each of these substances had a bitterness, expressed on a molar basis, equal to 20 per cent of that of quinine dihydrochloride. Neohesperidin and pinocembrin 7-β-neohesperidoside had approximately 10 per cent of the bitterness of naringin, and at this relatively low concentration, neohesperidin was capable of imparting bitterness to fruit.

The flavanone part of the molecule apparently contributes relatively little bitterness to the molecule as a whole; it thus follows that the sugar moiety is the structural part of the molecule that is responsible for taste sensations. However, in many glycosides, the presence of neohesperidose is not an essential factor for bitterness. The compounds listed in table 158 show that there are a number of bitter phenolic glucosides with no rhamnose in the molecule. Naringenin 7-β-D-glucoside is approximately 33 per cent as bitter as naringin (the parent glycoside); and hesperetin 7-β-D-glucoside has approximately the same bitterness as neohesperidin (the parent glycoside). Horowitz drew attention to the fact that bitterness was increased or maintained by attaching an α-L-rhamnosyl residue to the C2 hydroxyl group of D-glucose, but if the attachment was made to the C6 hydroxyl, bitterness was determined to be entirely eliminated.

Some Phenolic Substances of Grapefruit. In studying the phenolic substances of grapefruit, Maier and Metzler (1967*b*) isolated and identified, from extracts of mature grapefruit endocarp and peel, the principal aglycones after enzymic hydrolysis of the glycosides and esters. Glycosides or glucosides are usually widely distributed in the plant kingdom. They are derivatives, chiefly ethers, of hexose or glucose combined with various hydroxy compounds, such as alcohols, phenols, etc. On hydrolysis with dilute hydrochloric acid, the glycosides are decomposed, forming glucose or other hexoses and the corresponding hydroxy compound called aglycone. Until recent times, naringin and rhoifolin were the only phenolic substances isolated from the grapefruit endocarp, but with the development and perfection of chromatographic techniques, extracts of grapefruit endocarp show that other phenols remain to be identified. It is well known from the existing data that excessive bitterness in grapefruit is a limiting factor in its commercial acceptance. Therefore, Maier and Metzler made a comprehensive study of the phenolic constituents of grapefruit, and subsequently correlated and postulated a biosynthetic relationship of those constituents with the bitter flavanones.

TABLE 157

TASTE AND APPROXIMATE RELATIVE BITTERNESS OF PHENOLIC RHAMNOGLUCOSIDES AND AGLYCONES.*

Compounds	Taste	Molar concentrations of isobitter solutions	Relative bitterness†
Hesperetin	Slightly sweet	...	...
Eriodictyol	No taste	...	...
Naringenin	No taste	...	...
Pinocembrin	No taste	...	...
Phloracetophenone	Bitter	...	...
Hesperidin	No taste	...	...
Eriocitrin	No taste	...	...
Naringenin 7-β-rutinoside	No taste	...	...
Isosakuranetin 7-β-rutinoside	No taste	...	...
Phloracetophenone 4′-neohesperidoside	Bitter	1×10^{-3}	1
Phloracetophenone 2′-methyl ether 4′-neohesperidoside	Bitter	...	...
Phloracetophenone 2′,6′-dimethyl ether 4′-neohesperidoside	Bitter	...	...
Pinocembrin 7-β-neohesperidoside	Bitter	5×10^{-4}	2
Neohesperidin	Bitter	5×10^{-4}	2
Neohesperidin oxime	Bitter	...	...
Naringin	Bitter	5×10^{-5}	20
Poncirin	Bitter	5×10^{-5}	20
(Quinine dihydrochloride)	Bitter	1×10^{-5}	100

*From Horowitz (1961, 1964).
†On a molar basis, with quinine dihydrochloride taken as 100.

TABLE 158

TASTE AND APPROXIMATE RELATIVE BITTERNESS OF PHENOLIC GLUCOSIDES AND AGLYCONES.*

Compounds	Taste	Relative bitterness†
Phloracetophenone 4′-β-D-glucoside	Bitter	..
Sakuranetin 5-β-D-glucoside (sakuranin)	Bitter	..
Sakuranetin	No taste	..
Hesperetin 7-β-D-glucoside	Bitter	2
Naringenin 7-β-D-glucoside (prunin)	Bitter	6
Naringenin 5-β-D-glucoside (salipurposide)	No taste‡	..
Phloridzin	Bitter	10
Phloretin	Slightly sweet	..
Asebotin	Bitter	..

*From Horowitz (1961, 1964).
†On a molar basis, with quinine dihydrochloride taken as 100 (see table 157).
‡Data from Charaux and Rabaté (1931).

Naringin is the major, but not the only, phenolic constituent that is synthesized in growing and developing grapefruit. Maier and Metzler (1967*a, b*) isolated and identified the aglycone forms of the naturally occurring glycosides and esters of these phenols. In their first paper on the phenolic substances of grapefruit, Maier and Metzler (1967*a*) isolated and identified dihydrokaempferol from grapefruit where it had been shown to co-exist with naringenin and kaempferol. Horowitz (1961, 1964) reported the presence of the glycosides of naringenin and kaempferol in grapefruit (*Citrus paradisi* MacFad.).

Figure 105 shows the structural formula of 17 phenols which were identified and isolated from extracts of grapefruit endocarp after enzymic hydrolysis of

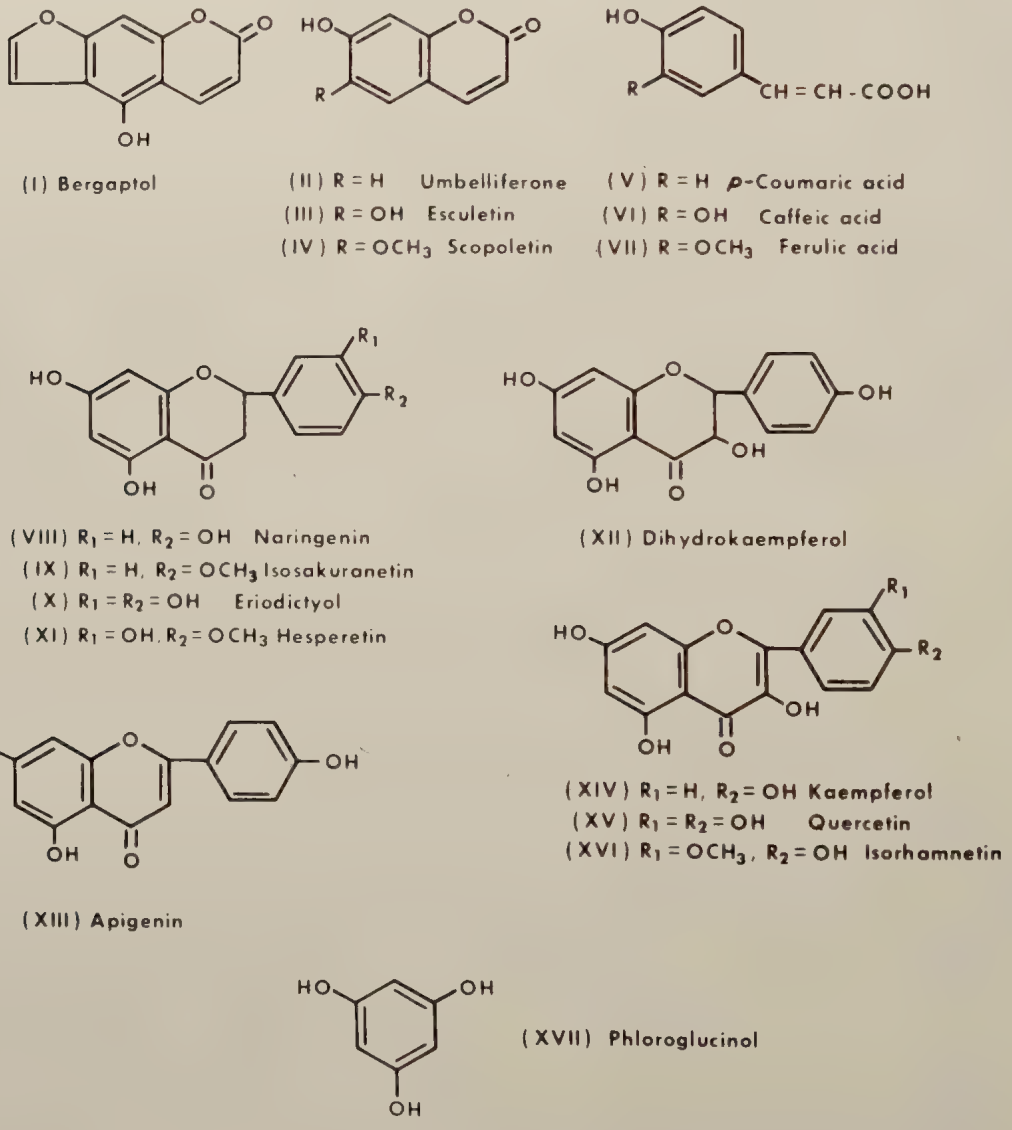

(I) Bergaptol
(II) R = H Umbelliferone
(III) R = OH Esculetin
(IV) R = OCH3 Scopoletin
(V) R = H p-Coumaric acid
(VI) R = OH Caffeic acid
(VII) R = OCH3 Ferulic acid
(VIII) R1 = H, R2 = OH Naringenin
(IX) R1 = H, R2 = OCH3 Isosakuranetin
(X) R1 = R2 = OH Eriodictyol
(XI) R1 = OH, R2 = OCH3 Hesperetin
(XII) Dihydrokaempferol
(XIII) Apigenin
(XIV) R1 = H, R2 = OH Kaempferol
(XV) R1 = R2 = OH Quercetin
(XVI) R1 = OCH3, R2 = OH Isorhamnetin
(XVII) Phloroglucinol

Fig. 105. The principal phenolic aglycones of grapefruit.
(From Maier and Metzler, 1967*b*.)

glycosides and esters (Maier and Metzler, 1967*b*) . The identifications of these aglycones were experimentally proved by direct comparison with known standard samples by means of TLC (thin-layer chromatography), ultraviolet spectra, paper electrophoresis, and color tests. All these phenolic aglycones except bergaptol occurred in grapefruit in the combined forms (glycosides and esters), and were capable of being hydrolyzed by enzymes. The presence of the free and combined forms was estimated by analyzing the endocarp extracts, before and after hydrolysis, with 2-dimensional TLC techniques.

The data reported in table 159 show the comparative amounts of phenolic constituents in the endocarp and peel of mature grapefruit. Maier and Metzler (1967*b*) reported that the peel contained the same phenols as the endocarp, but in different relative proportions. In both tissues, the phenolic constituents occurred in the combined forms, and they were converted to the free forms by enzymatic hydrolysis. Compositional differences, based on the total quantities present in the peel and endocarp of a typical grapefruit, showed that

TABLE 159

RELATIVE COMPOSITION OF THE ENTIRE ENDOCARP AND PEEL OF A TYPICAL MATURE GRAPEFRUIT.*

Occurring as glycosides and esters	Endocarp†	Peel†
Bergaptol	2	..
Umbelliferone	1	5
Esculetin	tr	tr
Scopoletin	4	1
p-Coumaric acid	3	2
Caffeic acid	4	2
Ferulic acid	5	5
Naringenin	10	50
Isosakuranetin	2	5
Eriodictyol	1	1
Hesperetin	2	1
Apigenin	3	7
Dihydrokaempferol	2	2
Kaempferol	2	tr
Quercetin	2	1
Isorhamnetin	2	tr
Phloroglucinol	2	14
Occurring as the free phenol	**Endocarp**	**Peel**
Bergaptol	3	2

*From Maier and Metzler (1967*b*).

†Numbers represent relative sizes and intensities of spots on thin-layer chromatography; "tr" equals "trace"; blank equals "absent." (See text of source citation.) Values can be converted from a "per-fruit" basis to a "per-unit weight of wet tissue" basis by multiplying endocarp values by 1 and peel values by 2.36.

phloroglucinol, umbelliferone, apigenin, and total flavanones existed in greater quantities in the peel than in the endocarp. On the other hand, endocarp had significantly more scopoletin and flavonol constituents than peel. The total phenolic component of the endocarp was more uniformly distributed within the individual constituents than it was in the peel. When the constituents were calculated on a wet-weight basis, all compounds except scopoletin, bound bergaptol, kaempferol, and isorhamnetin were present in greater concentrations in the peel than in the endocarp. Bergaptol was distinctive in that it occurred in the peel only in the free state.

After identifying 17 phenolic aglycones in grapefruit, Maier and Metzler (1967*b*) arranged these constituents according to a biosynthetic pathway, as graphically illustrated in figure 106. A close inspection of the diagram shows that p-coumaric acid was important in that all the compounds listed could be derived from this constituent. Consequently, these compounds showed a series of stable aglycone intermediates, starting from p-coumaric acid, to coumarin, furocoumarin, flavanone, flavone, and flavonol end-products. With respect to phenolic synthesis, they made the following statement.

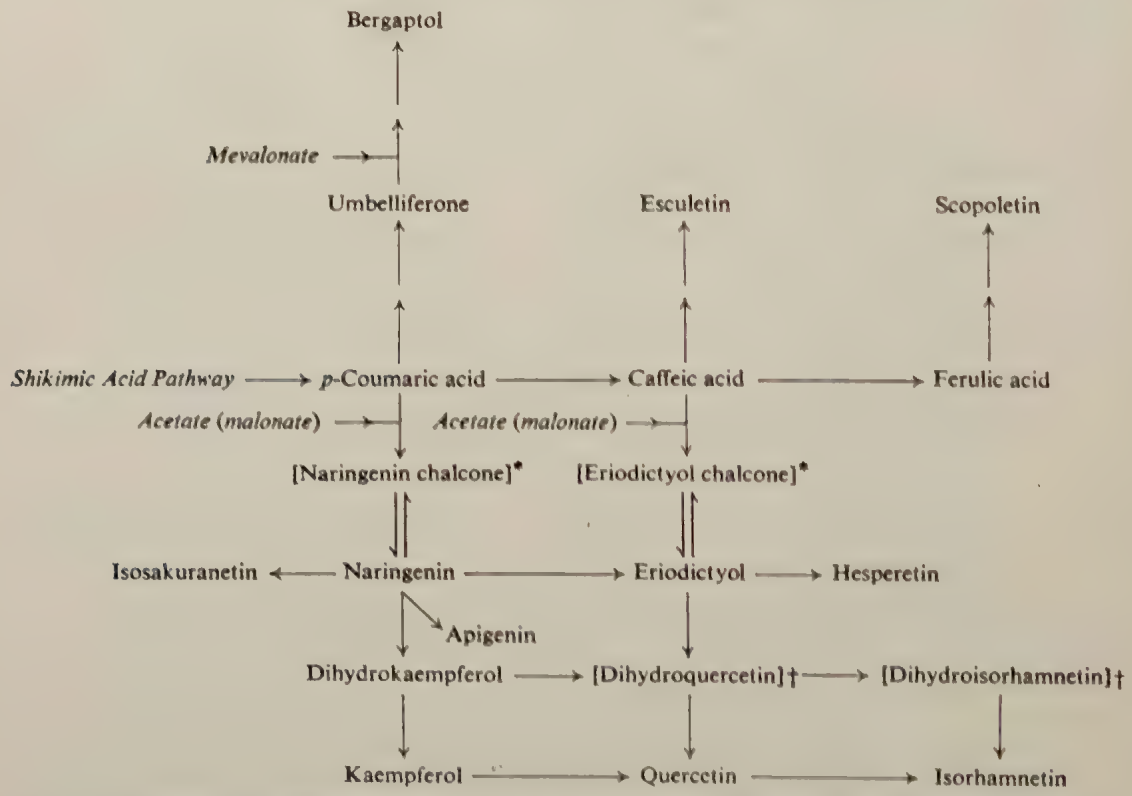


Fig. 106. Possible interrelationships of grapefruit phenolic compounds. Asterisks indicate that the compounds are not found. They would not be expected to be isolated because the equilibria favor the flavanone forms. Daggers indicate compounds not found, but the presence of which is suspected on the basis of preliminary evidence. (From Maier and Metzler, 1967*b*.)

At present, it is not known whether the phenolic constituents of citrus fruits are synthesized in place or are translocated into the fruit from the leaves. The wide array and apparent interrelation of the grapefruit phenolics reported here suggest that the fruit tissue does have the ability to synthesize or interconvert these compounds, at least starting from the early C_9 or C_{15} stages. The reported isolation of a flavanone synthease enzyme from the peel of several citrus fruits by Shimokoriyama (1957) also supports this view.

Limonoid Bitterness in Grapefruit. Limonin, a keto dilactone, has the possibility of existing in 2 monolactone forms: the A-ring monolactone and D-ring monolactone (Maier, 1969; figure 107). Maier and Margileth (1967) reported that the A-ring monolactone would be the naturally occurring form, but no data were obtained on the natural occurrence of the D-ring form.

It has already been mentioned that the bitter principle of grapefruit juice is chiefly ascribed to the flavonoid glycoside naringin, while the bitterness of navel orange comes from limonin (Higby, 1938; Emerson, 1948, 1949).

Maier and Dreyer (1965) reported the presence of limonin in grapefruit juice at levels above its taste threshold. Preceding their studies, limonin was only known to occur in citrus seeds and in the juice of sweet oranges. Their experimental data on grapefruit were obtained on mid- and late-season fruit

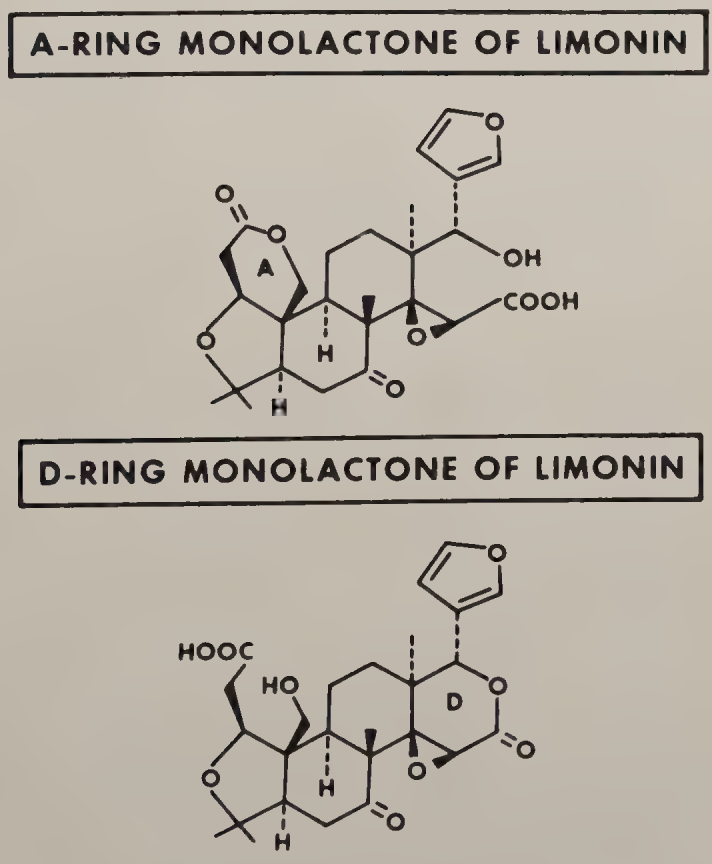


Fig. 107. Chemical structures of the 2 monolactone forms of limonin. (From Maier, 1969.)

because the degree of bitterness in the navel orange had been found to decrease significantly with maturity (Higby, 1938). On the other hand, in the late-season grapefruit tested, limonin was still present 6 months after the fruit had reached commercial maturity, in peel, endocarp, and seeds.

In the endocarp, limonin was found to be located principally in the carpellary membranes and the central vascular bundle (the spongy pith). The quantity that could be isolated from the endocarp tissues amounted to approximately 140 ppm (on a wet-weight basis). It has been suggested that the limonin content (or that of its precursor) in commercially prepared grapefruit juice probably entered the juice by diffusion from the pulp tissues during extraction and processing, the amount in the final product depending on the particular methods used in preparing the juice. In navel orange juice extraction, Higby (1941) noted that the bitter principle could be reduced by quickly separating the coarser tissue solids from the juice.

Maier and Margileth (1967) supplied a chemical basis for this process by showing that if maceration was avoided, the extraction of limonin A-ring monolactone was prevented from entering the liquid phase (the juice), which simultaneously facilitated the removal of the potential source of bitterness before limonin was formed. They discovered an enzyme in fruit-tissue extracts which, in acid solutions, changed the A-ring monolactone into limonin.

The activity of this enzyme appears to produce traces of limonin, which often occurs when extracting the monolactone from the fruit tissues. This enzyme may also partially convert the monolactone into limonin in orange and grapefruit juices during commercial juice production.

It is well known that in some years, navel oranges at various stages of commercial maturity will produce juice that will not become bitter after standing or processing. In their experiments on navel oranges at different stages of maturity, Maier and Beverly (1968) reported that the concentration of A-ring monolactone gradually decreased from the tissues as the oranges developed beyond commercial maturity, and that the limonin content of their extracted juice showed a corresponding decrease. This parallelism indicated, therefore, that navel oranges which contained no A-ring monolactone yielded juices which contained no limonin, and consequently no bitterness. As the authors indicated, their data confirmed that during the maturation of the fruit, the A-ring monolactone is metabolized by a fruit enzyme system.

Peel Oil. Camp *et al.* (1932) stated that color changes in extracted citrus juices were caused by the peel oil they contained, and that these changes were found to be about proportional to the amount of oil in the juice.

J. W. Sale (1947) reported finding a range of 0.001 to 0.018 per cent of essential oil in canned grapefruit juices from 4 states.

Scott (1941*a*) pointed out that the amount of peel oil that got into the extracted juice would depend on the type of extractor used. He gave the

figures quoted here on the oil recovered from extracted juice, stated as percentages by volume.

Reamed by hand	Trace–0.005
Belt-type extractor	0.016–0.017
Wheel-type extractor	0.005–0.016
Perforated cylinder	0.050–0.086
Fruit grated and pressed	0.075

Scott reported that small amounts of peel oil in the juice tended to improve its flavor, but that under unfavorable storage conditions, excessive oil eventually gave the juice a distinct turpentine-like flavor. This was probably owing to the deterioration of the terpene fraction of the oil.

Before 1948, the amount of peel oil in the extracted juice was determined by a distillation method suggested by Scott, which took about 90 minutes.

TABLE 160

SUMMARY OF THE VOLATILE OIL CONSTITUENTS OF GRAPEFRUIT JUICE.*
(Stated as milligrams per kilogram of juice.)

Constituents	Fresh	Freshly canned	Stored canned
Total oil (not including furfural)	20.92	26.00	27.59
Limonene	15.71	17.70	11.17
α-Pinene	tr	tr	tr
α-Caryophyllene	0.10	0.10	0.12
β-Caryophyllene	1.40	1.40	0.87
$C_{15}H_{24}$	0.11	0.11	0.14
$C_{15}H_{28}$	tr	tr	None
Low-boiling hydrocarbons	tr	tr	tr
N-Methyl methyl anthranilate	tr	tr	None
$C_{13}H_{15}N$	tr	tr	None
Caryophyllene oxide	0.80	0.80	0.27
Citral	~0.1	~0.1	~0.1
Carvone	~0.1	~0.1	~0.1
$C_{15}H_{26}O$	0.19	0.23	0.54
Linaloöl	0.16	0.23	0.08
$C_{15}H_{22}O$	0.45	0.42	0.64
Carveol	0.30	0.30	0.27
α-Terpineol	0.03	0.88	2.02
3-Hexene-1-ol	0.12	None	None
Geraniol	~0.05	~0.05	None
Linaloöl monoxide	0.37	2.03	8.95
$C_{12}H_{20}O_2$	0.20	0.20	0.20
Polyoxygenated compounds	0.40	0.86	0.97
Oxides	0.32	0.32	0.32
Furfural	None	tr	8.20

*From Kirchner and Miller (1953).

However, Burdick and Allen (1948) devised a turbidimetric method that reduced the time to 7 minutes.

In studying the flavoring materials of fresh and canned grapefruit juices, Kirchner and Miller (1953), in California, determined the volatile oil constituents of the juices. The volatile oils that they obtained were derived from peel oil incorporated in the juice by the method of extraction and from the oil naturally present in the juice vesicles—those that normally occur in commercially canned grapefruit juice. The data of table 160 show the comparative amounts of the different constituents found in the oil fractions of fresh, freshly canned, and stored canned grapefruit juice. Geraniol, $C_{13}H_{15}N$, and $C_{15}H_{28}$ were found in the fresh and freshly canned juices, but the alcohol 3-hexene-1-ol was found only in the fresh juice. The freshly canned juice had only a trace of furfural, but the stored canned product contained a significant amount.

The most notable changes found in the constituents were a decrease in limonene and an increase in linaloöl monoxide, α-terpineol, and furfural.

The data in figure 108 show the relative amounts of hydrocarbon and non-hydrocarbon constituents of the volatile oil fraction that occurred in fresh juice, freshly canned juice, and canned juice stored for 4 years. Kirchner and Miller (1953) concluded that the major changes in the volatile oil fraction as a result of heating and storage were increases in the oxygenated compounds, with corresponding decreases of the hydrocarbon constituents.

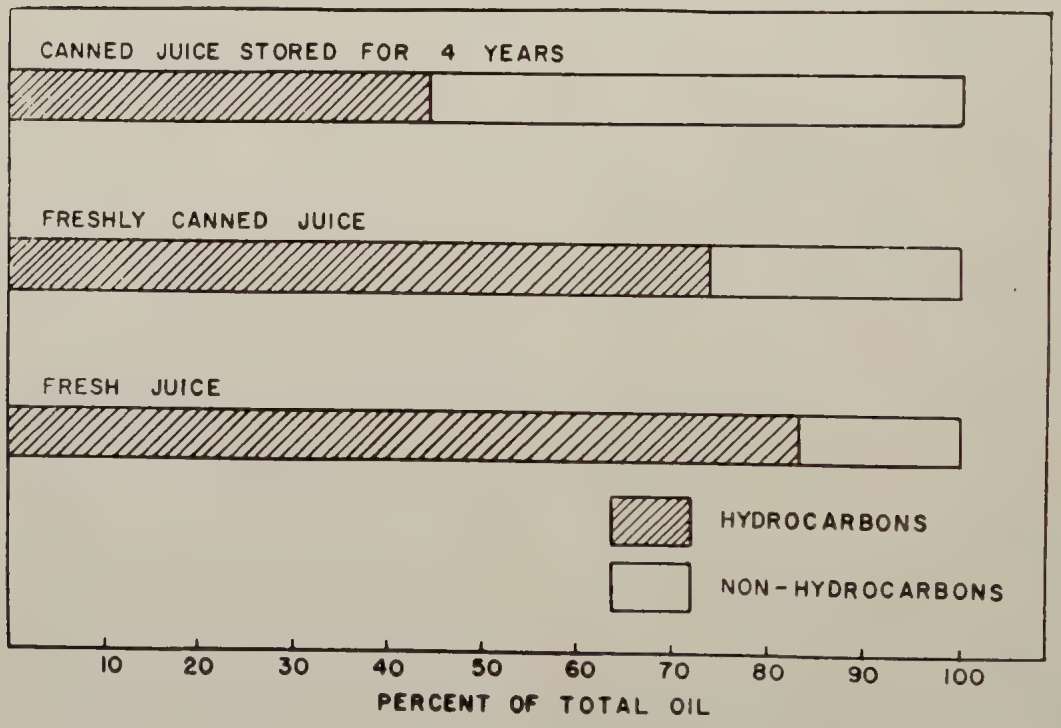


Fig. 108. Volatile oil from grapefruit juice. (From Kirchner and Miller, 1953.)

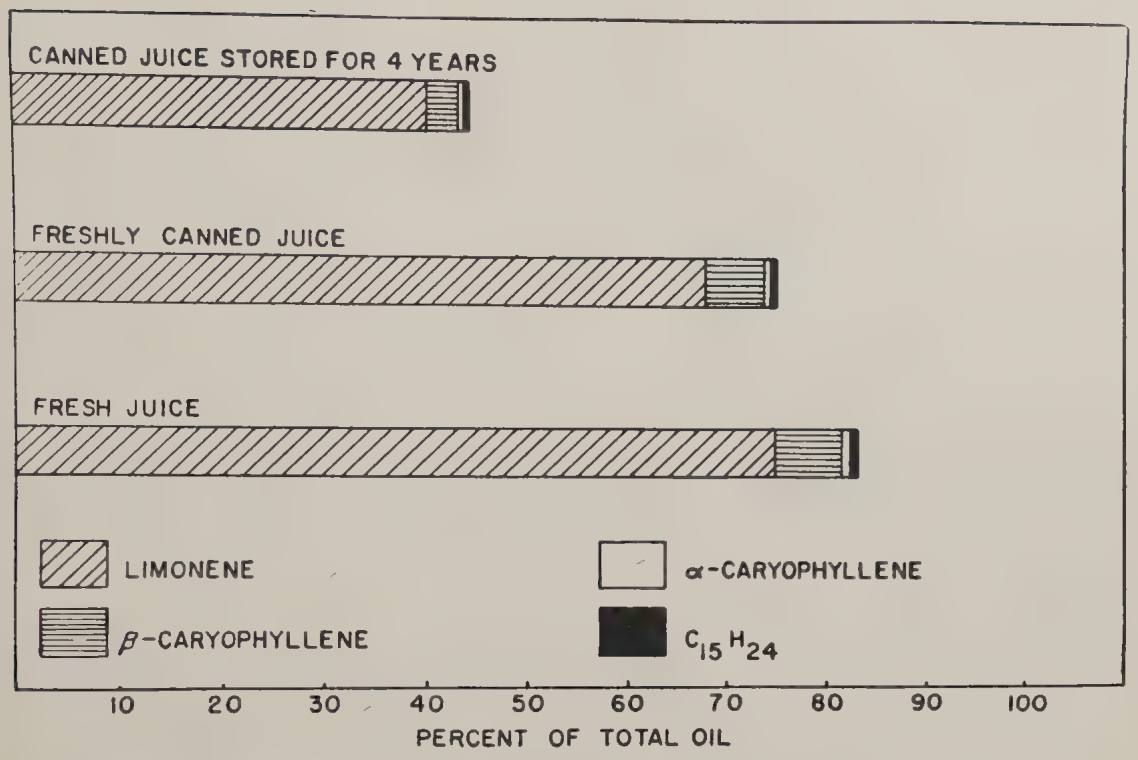


Fig. 109. Changes in the major hydrocarbon components of the volatile oil of grapefruit juice. (From Kirchner and Miller, 1953.)

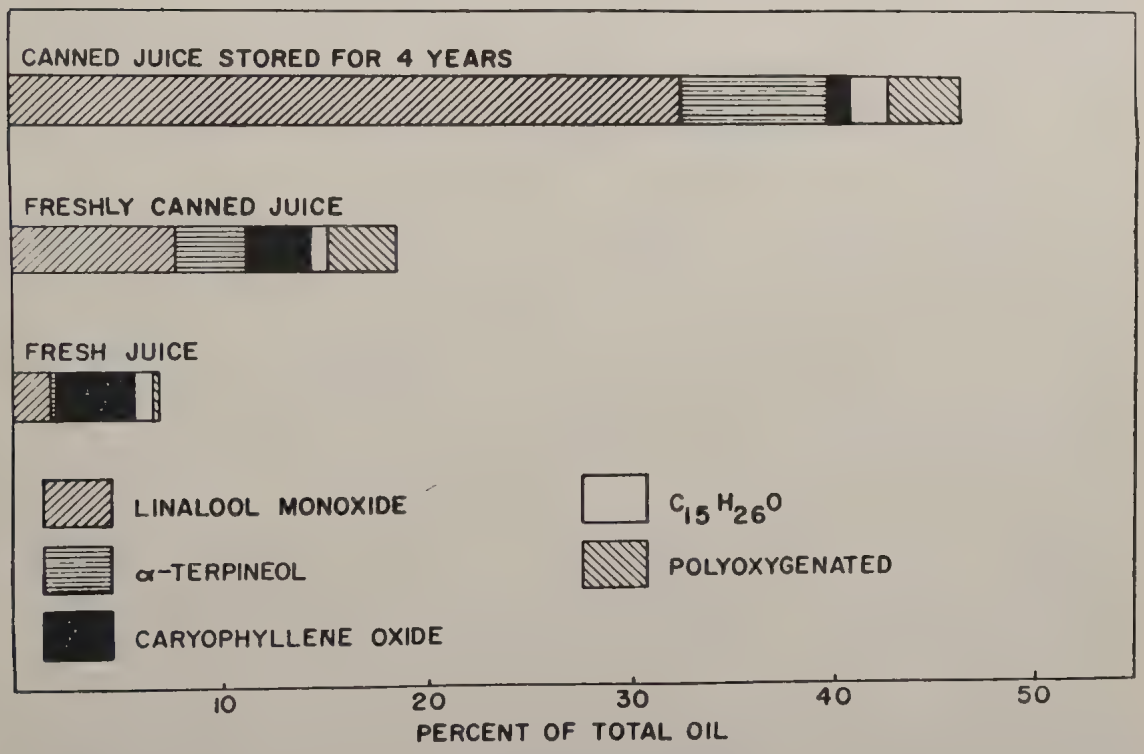


Fig. 110. Significant changes in the oxygenated components of the volatile oil of grapefruit juice. (From Kirchner and Miller, 1953.)

Figure 109 shows the changes in the major hydrocarbon compounds of the volatile oil fraction of grapefruit juice. It can be observed that the concentrations of limonene and β-caryophyllene were markedly reduced in the canned juice that was heated and stored for 4 years.

In the oxygenated portion of the volatile oil fraction of grapefruit juice, Kirchner and Miller (1953) observed the large increases in linaloöl monoxide, α-terpineol, and $C_{15}H_{26}O$ fractions in both the freshly canned juice and in canned juice stored for a 4-year period (figure 110).

Kirchner *et al.* (1953) isolated and identified the volatile water-soluble constituents from fresh juice, freshly canned juice, and stored canned juice (table 161). Note that the stored juice had a marked increase in methanol, acetic acid, and furfural, and the freshly canned juice had a small quantity of volatile acids and a trace of furfural. In the 2 types of canned juice, the volatile acids consisted of acetic acid and 2 unsaturated acids, listed as acid A and acid B, with the empirical formula $C_6H_8O_2$.

Kirchner and Miller (1953) also reported the results of taste studies on grapefruit juice with added amounts of various volatile oil constituents (table 162). In comparison with the same juice without the addition of oil, linaloöl monoxide, α-terpineol, and furfural produced taste differences that were considered by the panel to be undesirable.

Scott (1941*a*, *b*) called attention to the standards given below, set up by the United States Department of Agriculture in October, 1939, concerning the maximum recoverable oil permissible in commercial grapefruit juice.

1. U. S. grade A (fancy): Not over 0.014 per cent by volume.
2. U. S. grade C (standard): Not over 0.020 per cent by volume.
3. Off-grade (substandard): Fails to meet these requirements.

Microbiological Spoilage in Citrus Juices. There are 3 types of spoilage in fruit and fruit products: (1) owing to changes caused by the physical and chemical reactions occurring in the system, (2) enzymatic reactions, and (3) owing to changes caused by the presence of microorganisms. In this section, special emphasis will be placed on the third factor: spoilage of citrus juices by microorganisms. No attempt will be made to give an exhaustive analysis of the voluminous literature on the biological spoilage of citrus juices and concentrates. The chief purpose here is to give a résumé of the microbiological problems involved in the commercial processing of citrus concentrates by reviewing important pieces of published research which have contributed significantly to a better understanding of the behavior of the microorganisms involved.

Patrick and Hill (1959) published an extensive summary on this subject. Although most of their experimental results were obtained with oranges, the data are nevertheless highly applicable to similar problems in grapefruit.

Fruit juices show significant differences in their rates of deterioration in storage. With respect to their commercial quality, this deterioration is concerned chiefly with undesirable changes in flavor and appearance rather than with changes in nutritive value. Since fruit juices are composed of the cellular constituents of the fruit, including the accompanying enzymes, it is not surprising that these highly reactive materials undergo chemical changes that produce juices with off-flavors and chemical instabilities.

On the other hand, spoilage of fruit juices and products can also be caused by a wide variety of microorganisms, including bacteria, molds, and yeasts. The types of organisms that can produce spoilage in a given product are related to its chemical composition and its hydrogen ion concentration. Acid fruits with pH values of 4.5 or less are highly suitable for the growth of yeasts and molds, while acetic and lactic acid bacteria are exceptional in that they

TABLE 161

SUMMARY OF THE VOLATILE WATER-SOLUBLE CONSTITUENTS OF GRAPEFRUIT JUICE.*
(Stated as milligrams per kilogram of juice.)

Constituents	Fresh	Freshly canned	Stored canned
Acetaldehyde	1.45	0.33	0.6
Acetone	None	None	0.1
Furfural	None	tr	8.2
Ethyl alcohol	400.00	400.00	460.00
Methanol	0.2	0.2	23.0
Acetic acid	None	1.9	23.3
Acid A ($C_6H_8O_2$)	None	4.8	2.9
Acid B ($C_6H_8O_2$)	None	1.9	1.6
Acid C	None	tr	None
Hydrogen sulfide	tr	None	tr

*From Kirchner *et al.* (1953).

TABLE 162

TASTE STUDIES ON GRAPEFRUIT JUICE BY THE TRIANGULAR TASTE TEST.*

Oils added	Concentrations (mg/kg of juice)	Number of correct judgments out of 18	Comparisons with fresh juice (+4 to −4)	Panel comments
Linaloöl monoxide	90	17	−1.5	"Perfumy"
α-Terpineol	20	18	−2	"Musty"
Furfural	90	13	−3	"Burnt"
Linaloöl monoxide	9	13	−3	.....
α-Terpineol	2	13	−3	.....
Furfural	8	13	−3	.....

*From Kirchner and Miller (1953).

can tolerate a high concentration of hydrogen ions. Important as acidity is to microbiological activity, it should be noted that it is not the sole factor that limits the survival of microorganisms.

With the development of scientific methods for the conversion of citrus fruits into juices and other products, it was to be expected that both the sources and varieties of contaminating organisms would be increased. Patrick and Hill (1959) drew special attention to the fact that successful production of high-quality products begins with strict enforcement of sanitary regulations in the initial stages of the process. Citrus products prepared from fruit with high microbial contamination cannot benefit, in increased quality, by rigid plant and processing sanitation. In other words, it is difficult to produce a high-quality product from inferior fruit.

The sources and kinds of microorganisms that occur in fruit-processing plants are the same that are found with orchard-picked fruit that carries the common organisms that are normal to the season and location in which the fruit is grown. This statement is substantiated by the experiments of Teunisson and Hall (1949), who investigated the bacteria from citrus-processing operations in relation to orange juice quality, and reported that cultures common to citrus-processing plants contained the usual contaminants of soil, untreated surface water, and dusty air. The exterior contamination of the fruit will show considerable variation if it has had previous contact with the ground, or has been infested with organisms in airborne dust.

Citrus fruits should be selected, sorted, and cleaned before the juice is extracted. This problem has received considerable research emphasis over the years, stimulated by the continued increase in production of citrus fruit concentrates and other juice products. Beisel *et al.* (1950) emphasized the importance of washing the fruit with detergents that had been renewed intermittently during a day's operation. As a result of washing, the counts of microorganisms on the peel surface were reduced from 1 million organisms per cm^2 to 38,000 per cm^2.

Patrick (1949) also determined the population density of microorganisms on the peel of washed and unwashed citrus fruits. In any 1 test of 24 unwashed peel samples, he found a maximum of 210,000 microorganisms, with a mean value of 22,476 per cm^2. Corresponding figures for any 1 test of 19 samples of washed peel gave a maximum population of 18,880, with a mean value of 2,595 organisms per cm^2. The thoroughness of the washing procedures is indicated by the fact that 10 of the 19 samples contained 10 organisms per cm^2.

Murdock and Brokaw (1957*a*, *b*), in studying the specific sources of contamination in frozen orange concentrates, determined the effects of methods of handling the fruit and of extracting and concentrating the juice on the microbial content of the end product. Their results showed that the microflora determined on the surfaces of oranges, from bins to extractors, were the

sources of contamination. In the cleaning process, the brush washer in conjunction with a chlorinated water rinse was highly successful in reducing this surface condition. Juices extracted from defective fruits, which were usually highly contaminated by microorganisms, readily inoculated those extracted from healthy oranges. The authors also presented data to show that direct microscopic examinations and diacetyl determinations on the juices in process were good indicator methods for detecting microbial activity during periods of bacterial build-up.

The diacetyl test was first suggested by Hill *et al.* (1954) as a method to detect the growth of microorganisms in citrus juices and concentrates. The diacetyl values are directly related to the concentrations of both acetylmethylcarbinol and diacetyl in citrus products. These chemical compounds are the results of metabolic processes of acid-tolerant bacteria, such as *Lactobacillus* and *Leuconostoc*. The so-called "buttermilk flavor" which occurs in citrus fruit products has been experimentally associated with a certain concentration of diacetyl. The presence of acetylmethylcarbinol causes no off-flavor in citrus concentrates, but it may be associated with microbial activity.

Hill and Wenzel (1957) , in Florida, determined the frequency distribution of diacetyl values from 435 commercial samples of frozen concentrated orange juice collected twice monthly from 24 plants during the 1953–54 and 1954–55 seasons. In the reconstituted juices, the diacetyl values varied from 0.1 to 3.3 ppm; and most noteworthy was the fact that 88 per cent of the samples had diacetyl values of 1 ppm or less. The taste panel rated only 5 of the 435 samples as poor in quality because of "buttermilk" flavor. The diacetyl values reported for these 5 samples of concentrate were 1.2, 2.6, 3.1, 3.2, and 3.3 ppm, which values were considerably above the 1 ppm. These data seem to indicate that under strict sanitary operating practices, together with quality-control measures, such as diacetyl tests and direct microscopic counts, the consumer rarely encounters the "buttermilk" off-flavor in commercial frozen orange concentrates.

In observing many commercial samples of frozen orange concentrates, Hill *et al.* (1960) noted that the diacetyl values for juice products prepared from late-season Valencia oranges had higher diacetyl values than from those harvested early in the season. These observations led to the generalization that the differences in diacetyl contents of freshly extracted citrus juices from early- and late-harvested fruit were probably owing to the variety and degree of maturity. This is graphically illustrated in figure 111 for Pineapple and Valencia oranges and Marsh grapefruit. The diacetyl contents of Pineapple orange juice increased during the sampling period from a minimum of 0.7 to a maximum of 4 ppm. The last sample of Pineapple orange juice had an acid content of 0.66 per cent, soluble solids:acid ratio of 19.8, and pH of 4.1. The relatively low bacterial count of 4,200 for this juice sample (plated on dextrose

agar) indicated that the high diacetyl value of 4 ppm came from sources other than bacterial growth.

The diacetyl values of Valencia orange juice samples were similar to those of the Pineapple orange. During the 28-week sampling period, the diacetyl values increased from 0.9 to 2.6 ppm. The bacterial count of the final juice sample was comparatively low, with 8,000 colonies per milliliter of juice.

Marsh grapefruit juice showed seasonal changes in diacetyl content from a low of 0.1 to a maximum of 2.8 ppm. The values for both Marsh and Duncan grapefruit juices attained levels unusually higher than those found earlier for sound fruit. The final juice samples from Marsh and Duncan had a bacterial count of 51,000 and 17,000 per milliliter, respectively. These relatively high counts did not produce off-flavors in the juice samples. Hill and his co-workers concluded from these data that the diacetyl values were derived chiefly from acetylmethylcarbinol. With the rising trends of the diacetyl curves, a high acetyl value of freshly extracted citrus juice might partially indicate fruit growth and maturity without measuring the quality of the juice in terms of flavor or microbial content.

When viewed in terms of total acidity, the degree of maturity of oranges and grapefruit is of considerable importance in the production of a concentrate with a low microbial count. If the concentrate is produced from overmature, low-acid fruit, it will frequently have a high bacterial count.

Faville and Hill (1951) determined the relationship between the citric acid content of the fruit and the number of microorganisms in the extracted juice

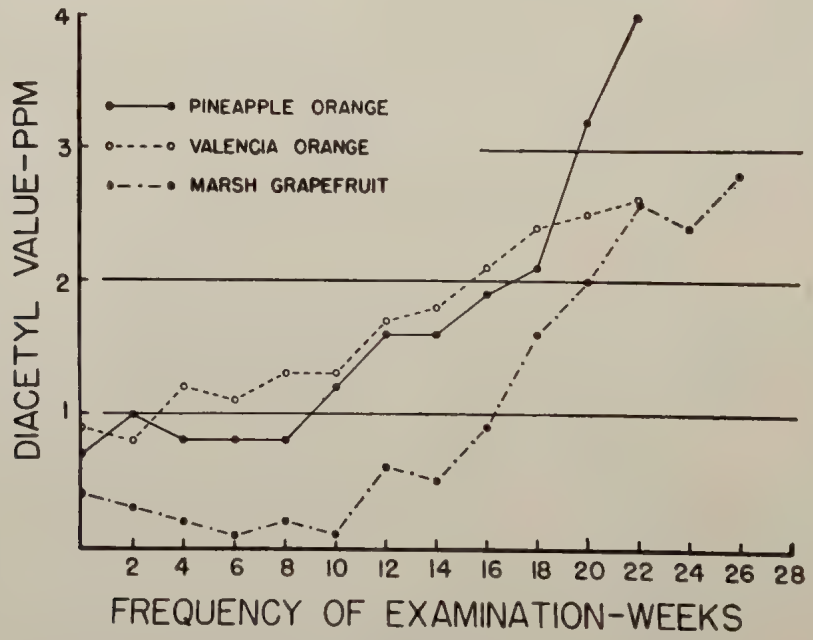


Fig. 111. Diacetyl values of Pineapple and Valencia oranges and Marsh grapefruit, picked and extracted at 2-week intervals. (From Hill *et al.*, 1960.)

(figure 112). Microbial counts obtained from orange juices with acid concentrations above 0.6 per cent were significantly low, showing fewer than 50,000 microorganisms per milliliter. Conversely, the microbial counts in juices with less than 0.6 per cent citric acid exceeded those in the higher-acid juices by 4- to 5-fold. The histograms shown for grapefruit juices are similar to those for orange, except that the former have microbial counts of a lower order of magnitude. In grapefruit juices with more than 1.6 per cent citric acid, the yeast and mold counts amounted to the total microbial counts of the cultures.

The survival of microorganisms in citrus juices and concentrates is markedly affected by their storage temperature and the concentration of solids in the product. In the experiments of DuBois and Kew (1951), the temperature factor was of special interest. They stored commercial packs of orange and grapefruit-orange concentrates at various temperatures, and subsequently determined, at intermittent periods, the population density of the microorganisms in the samples. About 88 per cent of the microorganisms were destroyed in cans stored at temperatures of –20, –8, zero, 5, 10, 15, 20, 25, 45, and 70° F. (–29, –22, –18, –15, –12, –9.4, –6.6, –3.8, 7.2, and 21° C.) for a 30-day period. Other experiments showed that 75 per cent of the microorganisms were destroyed in 7 days at 20 and 25° F., and 75 per cent in 15 days at 15 and 45° F. These researchers noted that with the rapid decrease in microbial

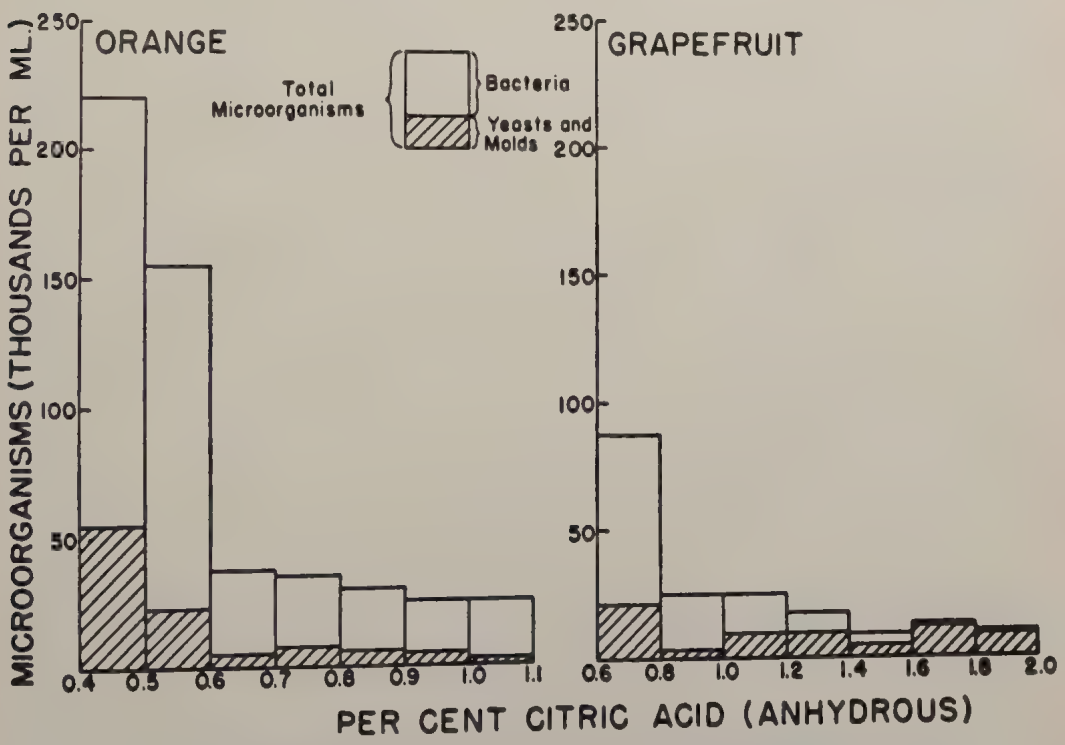


Fig. 112. Relationships between citric acid content and microorganisms in citrus juices. (From Faville and Hill, 1951.)

count, any changes occurring in the juices during storage would have to be attributed to factors other than microorganisms.

Moore *et al.* (1950), in studying the effects of storage temperatures on the microbial counts of citrus concentrates, stored 6 freshly packed concentrates: 2 grapefruit (Duncan and Marsh Seedless); 3 orange (Valencia, Hamlin, and Pineapple); and 1 tangerine (Dancy) for a 6-month period at various temperatures. Microbial counts were made on samples at intervals during the storage period. Figure 113 shows the trend in reduction of the average total microbial counts of the 6 packs at various temperatures. The curves representing concentrates stored at 32 and 40° F. (zero and 4.4° C.) showed a 90 per cent reduction in total microorganisms within the first month of storage, and after 6 months, the count leveled off to 96 per cent. Concentrates stored at

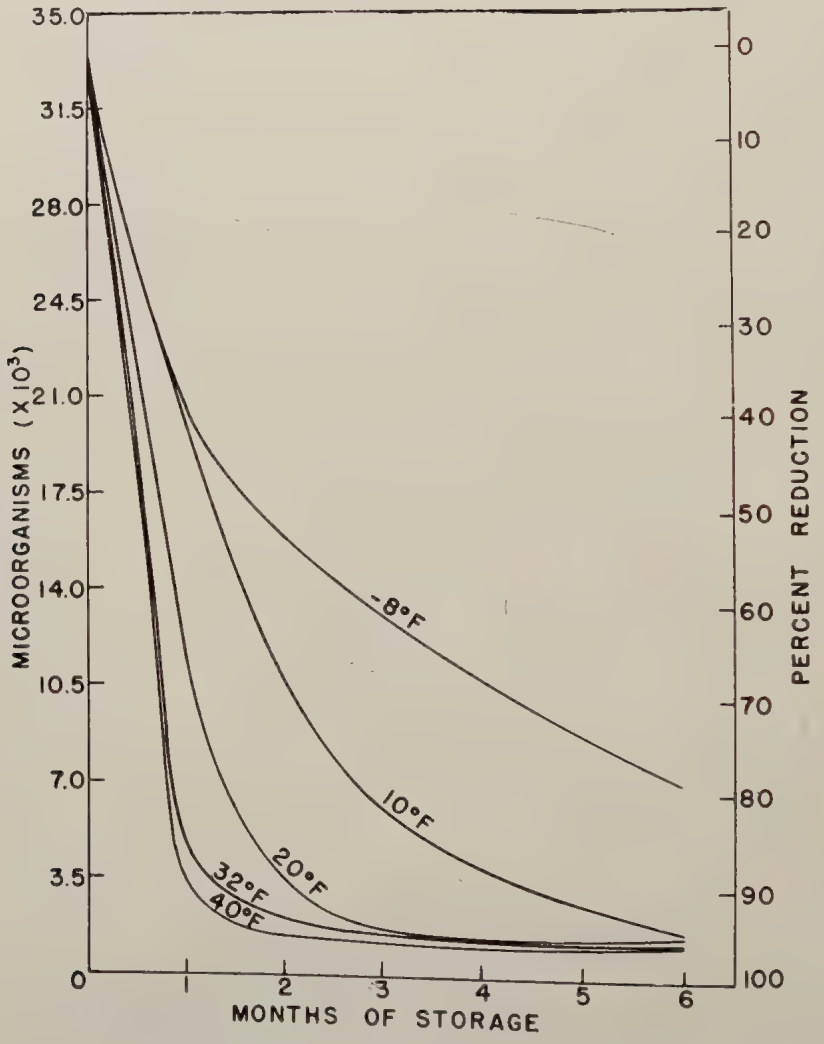


Fig. 113. Trends in reduction of average total microbiological counts of 6 lots of freshly packed citrus concentrates stored at different temperatures. (From Moore *et al.*, 1950.)

20° F. (–6.6° C.) showed a reduction in total count which closely approached, in 3 months, those concentrates stored at 32 and 40° F., and continued at this level for the ensuing 3 months. In concentrates stored at –8° F. (–22° C.), the total count decreased at a slower rate, with a gradual reduction to 80 per cent at the end of the 6-month storage period. The slope of the line of the 10° F. (–12° C.) concentrates fell between the curves plotted for –8 and 20° F. At the end of 6 months' storage, the reduction in total microbial count of the 10° F. concentrates amounted to approximately 95 per cent. It thus can be observed that low-temperature refrigeration reduces the rate of reproduction of microorganisms to a point where the metabolic products will not have reached a concentration sufficient to affect the flavor of the concentrates before they are consumed.

Rushing *et al.* (1956) investigated the influence of pH and juice concentration on the growth rates of *Lactobacillus* and *Leuconostoc* species in orange juice. These 2 genera are known to occur frequently in orange concentrates, and several strains of the organisms were identified with a type of spoilage known as "buttermilk off-flavor," which was associated with the presence of diacetyl in the products. Figure 114 shows the growth rates of these bacterial genera in 12° Brix juice at different pH values. The slope of the curve for *Leuconostoc mesenteroides* showed responses to changes in pH that were markedly different from those of the others. The highest growth rate found in this study was recorded for *L. mesenteroides* in 12° Brix orange juice at pH 4. This organism also showed a negative growth rate at 12° Brix and pH 3.4. Negative growth rates indicated a decrease in viable plate count as a result of unfavorable environmental conditions. This species was particularly sensitive to changes in pH. As the curves plotted for the 2 *Lactobacillus* species were close together and essentially parallel, they had about the same response to pH changes. In terms of growth rates, *Leuconostoc dextranicum* was more sensitive to pH change than the 2 *Lactobacillus* species.

Rushing and associates also noted that growth rates of all the organisms studied decreased with an increase in juice concentration (figure 115). *Lactobacillus brevis* and *Leuconostoc mesenteroides* exhibited negative growth rates at 42° Brix concentration. The curves of the 2 genera showed slight differences, but they formed approximately parallel lines. *Leuconostoc* species showed a greater decrease in their rates of growth than *Lactobacillus* species between 12 and 18° Brix concentrations. As the concentration increased above 37° Brix, *Lactobacillus brevis* exhibited the greatest decrease in growth rate. These authors indicated that generation time might be more useful than growth rate in interpreting microbial behavior in the plant. *Leuconostoc mesenteroides,* with its exceptionally high growth rate, had to remain in the evaporator at 12° Brix and pH 4 for 1.4 hour in order to double its population. Juice was concentrated very rapidly in the evaporator, and the total

retention time amounted only to a few minutes. Consequently, they noted that factors other than the growth rates of the organisms would have to be considered if sufficient numbers were produced to cause development of off-flavors in commercial evaporators.

Faville *et al.* (1951) reported that bacteria, and not yeasts, were the predominant organisms present in 42° Brix orange concentrates, and that a significant number of bacteria were killed in storage at zero Fahrenheit (–18° C.). They also observed that bacteria were killed at a faster rate in 42° Brix concentrate than in single-strength juice. Yeasts accounted for about 20 per cent of the total initial count of microorganisms.

Patrick (1949) reported that the storage of orange concentrate at zero Fahrenheit for 198 days reduced the number of spoilage organisms, and that

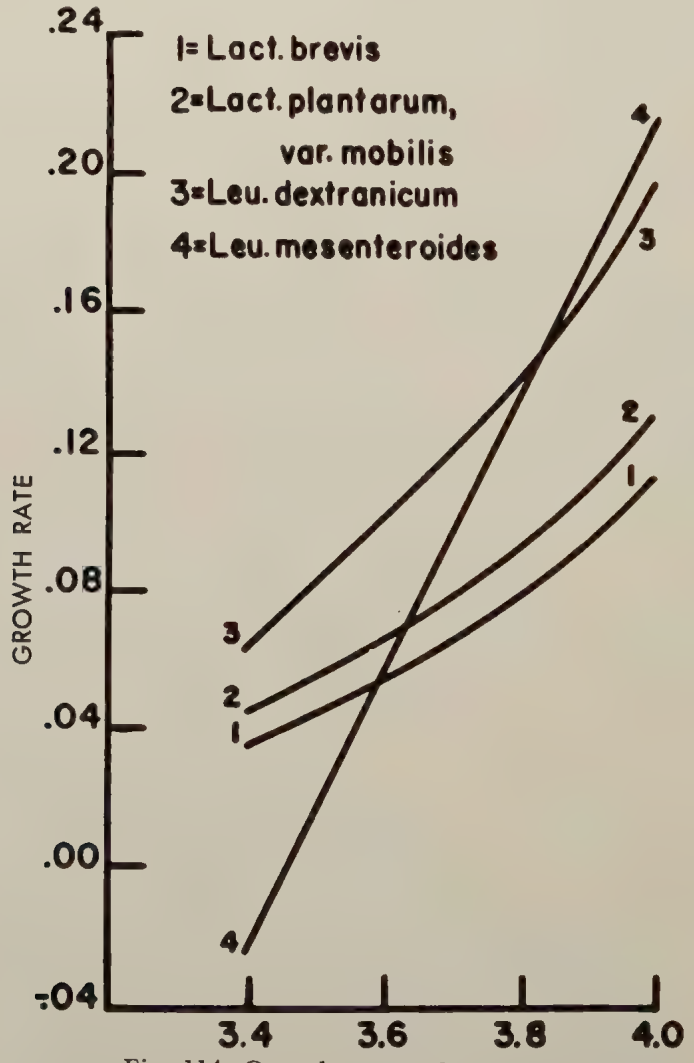


Fig. 114. Growth rates of *Lactobacillus* and *Leuconostoc* species in orange juice of 12° Brix at different pH values. (From Rushing *et al.*, 1956.)

yeasts predominated among the surviving organisms in concentrates stored at both zero and 42° F. (–18 and 5.5° C.).

Patrick and Huggart (1955), in studying the survival and growth of microorganisms in citrus concentrates, noted an accelerated rate of decline in microbial counts of 4- and 6-fold concentrates after the first month of storage at –8, 20, and 40° F. (–22, –6.6, and 4.4° C.). The rate of decrease of organisms

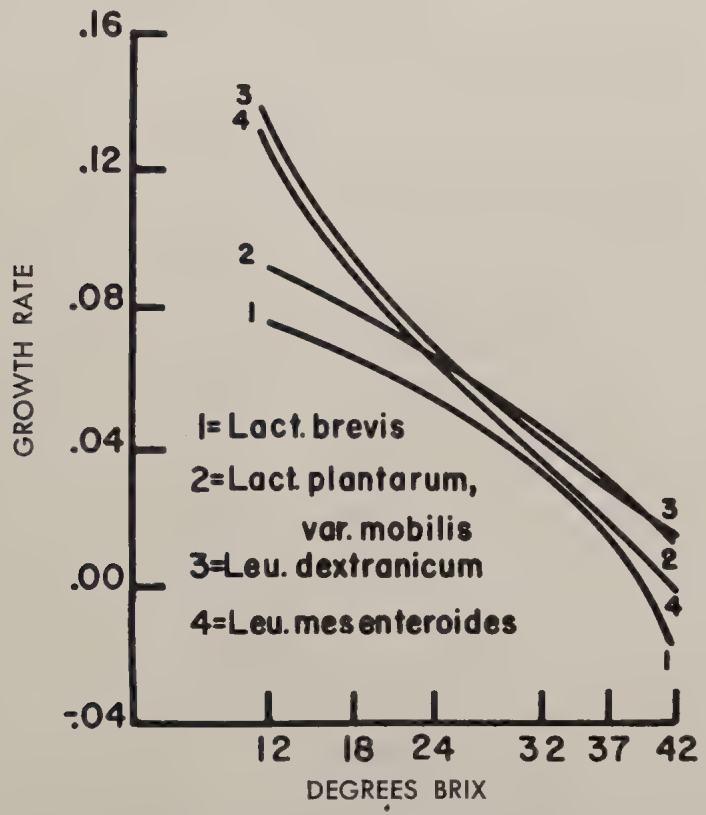


Fig. 115. Growth rates of *Lactobacillus* and *Leuconostoc* species in orange juice of pH 3.8 at different degrees Brix. (From Rushing *et al.*, 1956.)

at –8° F. was slower than at higher temperatures. The reduction in counts in all samples extended from 74 per cent in 1 pack stored at –8° F. to 99 per cent in concentrates stored at higher temperatures. Storage of concentrates at 60° F. (16° C.) resulted in fermentation, clarification, and gelation of the juices.

Wolford and Berry (1948*a*, *b*) and Wolford (1950) were among the earlier workers who studied the factors affecting the presence of coliform organisms in oranges and orange juice. In his studies, Wolford reported on the survival of these organisms in frozen orange juice stored at –10° F. (–23° C.) for a period of 43 weeks. The total count dropped rapidly during the first 6 weeks of the storage period. The subsequent rate of decline was less, but it followed

a logarithmic slope, indicating the presence of a mixture of sensitive and resistant forms. The survival of coliform bacilli in these frozen juice samples stored at –10° F. did not follow the trend observed in the total plate count. It was experimentally shown that organisms resembling *Escherichia coli* were obtained most frequently from 2 lots of juice prepared from soft fruit, while juice prepared from sound fruit in a clean plant contained none of these organisms. Various bacilli that met the definition for coliform remained viable in frozen juices stored at – 10° F. for 43 weeks. In the course of this experiment, 236 cultures of coliform organisms were isolated from 79 juice samples. Forty-six of these cultures resembled *E. coli;* 20 were intermediate forms (*E. freundii*); and 170 resembled species of *Aerobacter.*

Patrick and Wenzel (1959) reviewed the data on the occurrence of coliforms in citrus products, so that a better interpretation and application of the existing information could be obtained with respect to the sanitary significance of these organisms when present in the products. Their analysis indicated that coliform bacilli could be isolated from the external surfaces or juices of oranges collected from different localities. Coliform organisms were isolated not only from soft and damaged fruit, but they also appeared on fruit infested with red scale, and contamination could be carried by fruit flies. The coliforms on the surfaces might be sources of ultimate juice contamination.

Wenzel and Moore (1955) stated that the methods used to prepare fruit for extraction and the thermal stabilization procedures used to inactivate the enzymes before or during the concentration of citrus juices were undoubtedly destructive of many coliform types present in the final product, and that the excellent sanitary provisions then in practice were important with respect to the reduction of coliforms in the juices. Before the adoption of strict regulations for plant sanitation, various pieces of equipment used in juice processing had been sources of coliform organisms in citrus juices. From their analysis of the available published information, the foregoing authors concluded that the presence of coliforms in frozen citrus concentrates and other processed products had no sanitary significance. This statement is still borne out by the fact that millions of gallons of processed citrus juices are consumed annually without any deleterious effects on human health.

During the years of development of the commercial processing of citrus fruit, considerable research was published on the effect of storage on the survival of enteric and pathogenic organisms in citrus juices and concentrates. The presence of coliforms and enterococci in citrus juice products caused projects to be initiated to isolate these organisms, to obtain further data on their modes of entry and the sources of contamination, and to determine their effects on health.

Kaplan and Appleman (1952) isolated 76 strains of enterococci from commercial frozen orange concentrates; 63 were identified as *Streptococcus lique-*

faciens, which had the ability to liquefy gelatin, and 13 as *S. faecalis.* Enterococci were isolated from all cans, even in 0.1-milliliter samples of juice as inocula. Enterococci had a higher resistance to adverse environmental conditions than *Escherichia coli.* They noted the possibility of a relationship between enterococci and pollution in frozen foods.

Farraro and Appleman (1957) found enterococci in samples from 9 commercial brands of orange concentrate.

In studying the enterococcus-like organisms in citrus juices, Patrick and Hill (1958) confined their attention to identification of the streptococcus-like organisms in the juices which had possible enteric properties. They found that 13 of the cultures isolated from citrus concentrates survived the confirmatory test for these organisms; 7 of the cultures resembled *Streptococcus liquefaciens,* and 6 resembled *S. faecalis.* The authors were inclined to the view that streptococcus-like contamination of citrus concentrates would be associated with over-mature and low-quality fruit. It seems highly probable that these organisms survived from the orchard where the fruit was grown, and thus reflected the quality of the fruit processed.

The destruction of microorganisms in citrus juices by heat treatment is dependent upon the desired temperature to be attained by the juice and the time of exposure to this temperature. In heat sterilization, the higher the temperature, the shorter will be the time required to completely destroy the organisms. Sterilization of citrus juices can be accomplished at temperatures above those required for optimum growth of the organisms, the time of heating varying inversely with the temperature. High-temperature, short-time heat treatments of citrus juices kill microorganisms and inactivate enzymes with less change in flavor than the reverse process of low-temperature, long-time treatments.

Nolte and von Loesecke (1940*c*) investigated the types of organisms in Florida citrus juices that could survive high temperatures for short periods of time. They reported that the organisms which survived the heat treatments did not increase in numbers when reinoculated into canned orange or grapefruit juices. Thermophilic organisms were readily destroyed at relatively high temperatures (about 248° F. or 120° C.), and in some instances this required time periods as long as 35 minutes.

After studying temperature–time variables as factors in the destruction of microorganisms in citrus juices, Patrick and Hill (1957*a*) reported that *Lactobacillus plantarum* in single-strength Valencia orange juice at pH 3.6, 3.8, and 4.0 was destroyed by heating for 12 seconds at 155° F. (68° C.). Under similar experimental conditions, except at pH 4.0, *Leuconostoc mesenteroides* was destroyed in 6 seconds at 155° F. At pH 4.0, complete destruction of the organism occurred at 165° F. (74° C.) in 12 seconds. They inoculated juice samples (pH 3.8 and 4.0) with a mixed culture of organisms common to orchard-run

fruit, and subsequently heated the samples to 190° F. (88° C.) for 12 seconds. This temperature was insufficient to completely destroy the bacteria present.

Patrick and Hill (1957*b*) extended the foregoing studies to include the effects of heat treatments on the survival of microorganisms in 2-, 3-, and 4-fold concentrates prepared from orchard-run fruit, or with an unidentified mixed culture added to the concentrates at pH 3.9. The organisms were not completely destroyed in the 3 concentrates exposed to temperatures up to 185° F. (85° C.) for 12 seconds. They also noted that the number of organisms surviving the heat treatments decreased with increases in juice concentration. Samples heated to temperatures above 165° F. (74° C.) showed a definite reduction in the number of survivors, regardless of the concentration.

In contrast to the fact that many essential oils were thought to possess antimicrobial properties, Murdock *et al.* (1967) showed that orange oil was an excellent medium for microbial growth. A similar relationship held for the essential oils of other citrus types. These researchers isolated 4 different strains of medium-sized rods of varying morphology from orange serum agar plates. They observed that the growth rates of the isolates could be accelerated by the addition of orange oil emulsion to orange serum agar. Each of 4 aliquot portions of sterilized orange oil emulsion (15 minutes at 250° F. or 121° C.) was inoculated with 1 of the test organisms and incubated at 86° F. (30° C.). The emulsion was plated at periodic intervals, and the oil phase was analyzed by gas chromatography. All test organisms grew well when inoculated into sterile orange oil emulsion, and all strains except 1 produced α-terpineol.

The curves shown in figure 116 illustrate the microbial growth and production of α-terpineol in inoculated orange oil emulsions at various numbers of days during incubation. It can be observed that α-terpineol continued to increase even after the maximum survival point of the organisms. Murdock *et al.* (1967) suggested that as long as some microbial growth was sustained, α-terpineol would continue to be produced. This indicated that the concentration of terpineol continued to increase even after the organisms had reached their maximum population density. The second half of the growth curve shows that the organisms were dying faster than they were being reproduced, and that terpineol continued to be produced under these conditions. Since α-terpineol is a chemically stable compound, the concentration steadily increased during the incubation period.

The data presented in figure 116 strongly indicate that α-terpineol in orange oil is a metabolic product of bacterial growth, and in the absence of bacterial growth, its concentration in the oil remains approximately constant. These microorganisms grow in the aqueous or oil-free phase of the slurry and/or emulsion. It was noted that certain strains might have developed a tolerance for high concentrations of oil, but plate counts made of the finished oil were found, for all practical purposes, to be free of microorganisms.

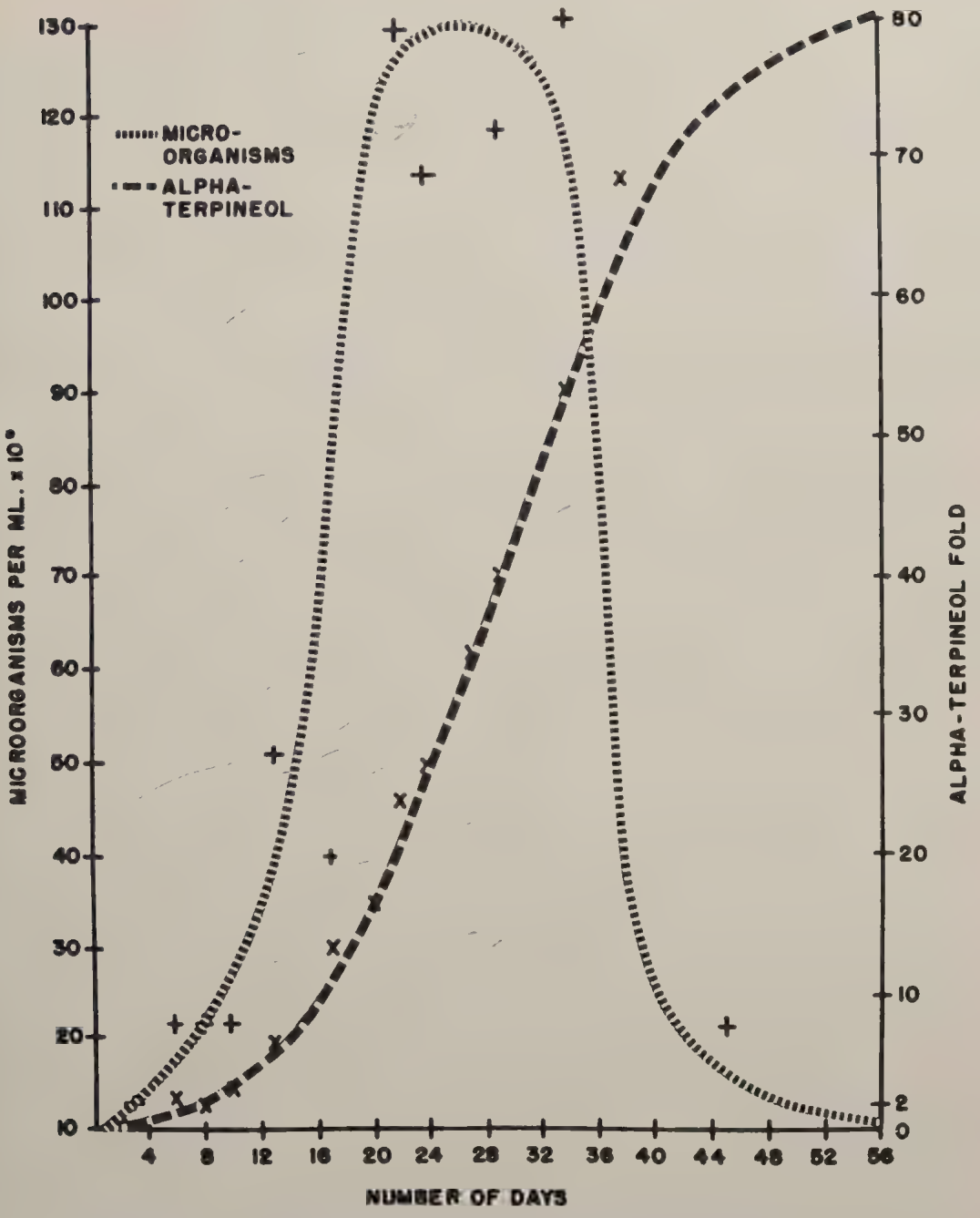


Fig. 116. Growth of microorganisms in orange-oil emulsion and increase of *alpha*-terpineol in orange oil. (From Murdock *et al.*, 1967.)

Factors That Affect Yield

MANY FACTORS that affect the production or yield of grapefruit have already been discussed in this chapter. See the discussions of fertilizers, irrigation, rootstocks, and freeze injury under their respective subheadings, starting on page 307. Variety, locality, climate, and type of soil are all factors related to yield, and there is a considerable amount of experimental data based specifically upon them. Their effects are reported in connection with the results of research on fruit quality, especially as it concerns juice composition and volume. The following text deals with a few additional matters in connection with yield, such as the selection of buds from high-yielding trees, age of trees and fruit, and hormones.

Friend and Yarnell (1940), based on the results of their tests with Marsh and Thompson grapefruit, all but 2 trials of which were budded onto sour orange, concluded: (1) that the use of budwood from normally vigorous trees of relatively low productivity was not likely to be reflected in the yielding capacities of the resultant trees; and (2) that strains of superior productive capacity were not to be expected through the exclusive use of budwood from trees known for their high yields. In contrast to these findings, Batchelor and Webber (1948, page 15) stated: "Thus, the policy of taking buds from high-yielding trees only is a safe one, and the only one that can be advocated as reliable."

Shamel (1943), as a result of extended experimentation on the nature and occurrence of bud variation, summarized the advantages of commercial bud selection in citrus fruit thus: (1) bud selection avoids the propagation of inferior and undesirable strains; (2) there is an opportunity to develop exceptionally heavy-producing and uniformly high-quality progenies, of which some may eventually be classified as distinct strains; (3) the discovery of desirable sources of bud supplies is possible; (4) the possibility exists of finding and testing some apparently superior strain originating as a bud variant; (5) to develop the recognition of the use of bud selection as a means of improving citrus production; and (6) the improvement in production of older orchards by top-working healthy trees of undesirable strains with buds from selected strains, or by replanting with nursery trees grown from selected buds.

Evidence obtained by Finch and McGeorge (1940) confirmed the findings of others that nitrogen, when properly applied, would play an important role in regulating the fruitfulness of grapefruit trees. In contrast to Innes (1946), these workers found that potassium or phosphorus applied separately, in combination with each other, or with nitrogen, seemed to have little effect on the yield of grapefruit. On the other hand, Innes, during the period from 1936 to 1946, found that potassium plus phosphorus not only caused an increase in yield, but also an increase in weight per fruit.

Fudge (1938, 1939), in Florida, concluded that magnesium deficiency was responsible for a decrease in yield of fruit. In this connection, Camp and Jeffries (1937), also in Florida, found that the seedless varieties of grapefruit had a higher yield than the seedy ones. They attributed the difference to magnesium deficiency in the foliage of the seedy varieties, the magnesium having been absorbed by the seeds.

Tree Age. Botts (1941), in Florida, made a study of the effect of tree age on yield of fruit. Production data for 6 years or more were available for about

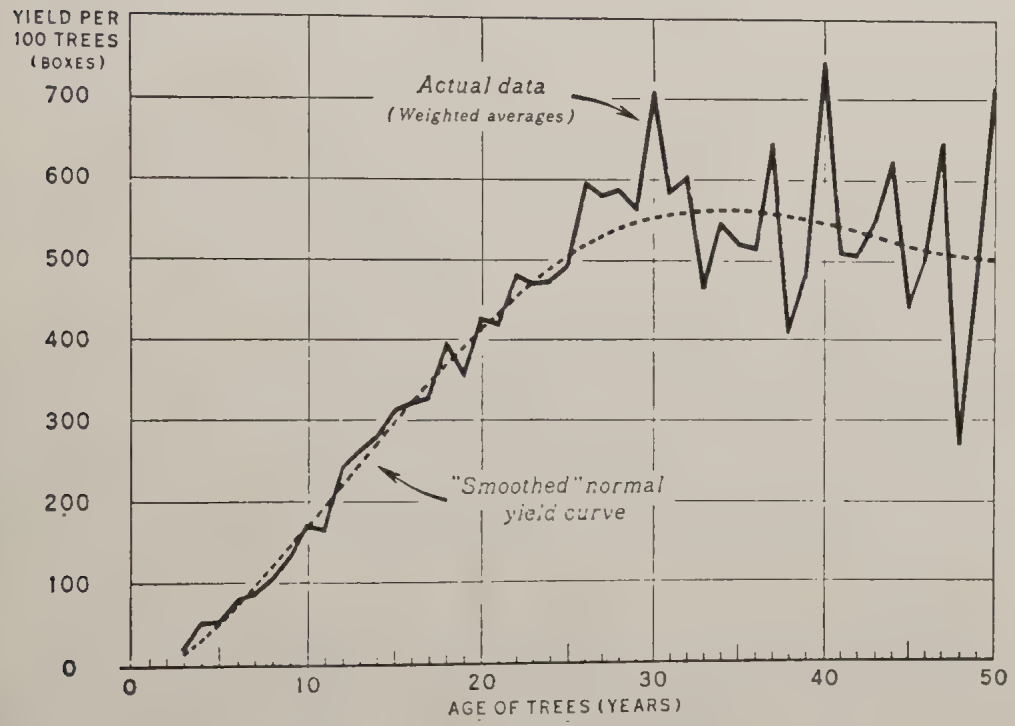


Fig. 117. Florida grapefruit: Yields per 100 trees by tree ages. The actual weight averages were computed with "smoothed" data. (From Botts, 1941.)

half of the groves checked, while records for varying shorter periods were available for the remainder. The results for grapefruit are shown in figure 117. It appears that yields of Florida grapefruit increased on the average until the trees reached about 34 years of age, after which their yield started to decline. More "smoothing" of the weighted averages was necessary at the greater ages (beyond 25) than was necessary at the earlier ones.

Husmann *et al.* (1962) developed experimental data showing that the trend in production of Marsh grapefruit trees generally increased up to 30 years of age, but a careful inspection of the production curve of these plots indicates that they reached their peak production in the vicinity of 28 to 30 years, and that thereafter the rate declined perceptibly (figure 118). Although the yield

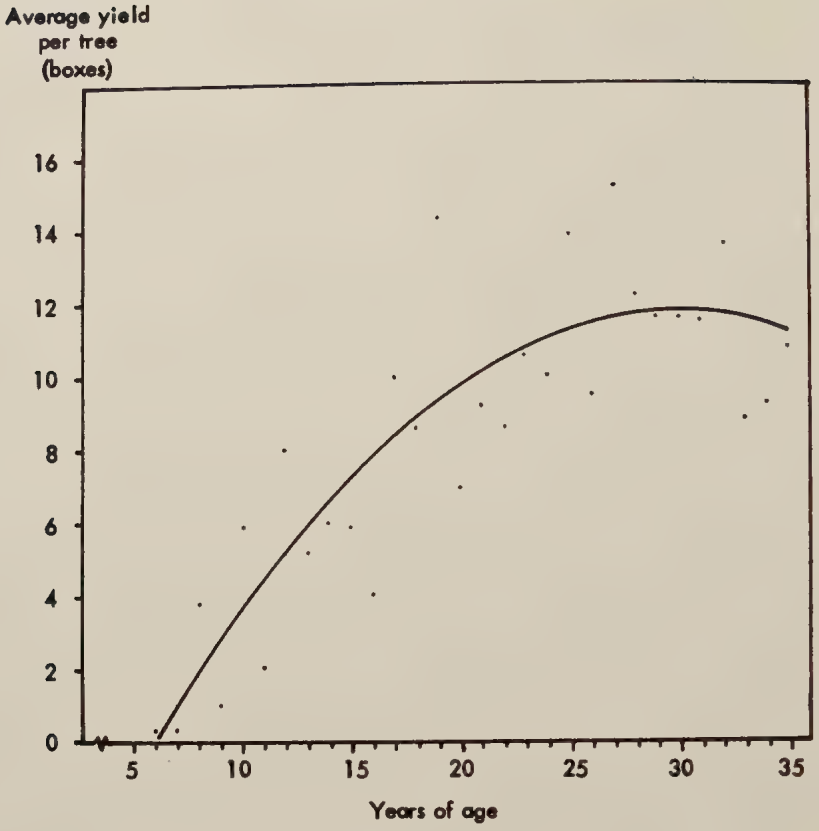


Fig. 118. Relationships between average yield per tree and tree age for Marsh grapefruit grown in southern Lake County, Florida. (From Husmann *et al.*, 1962.)

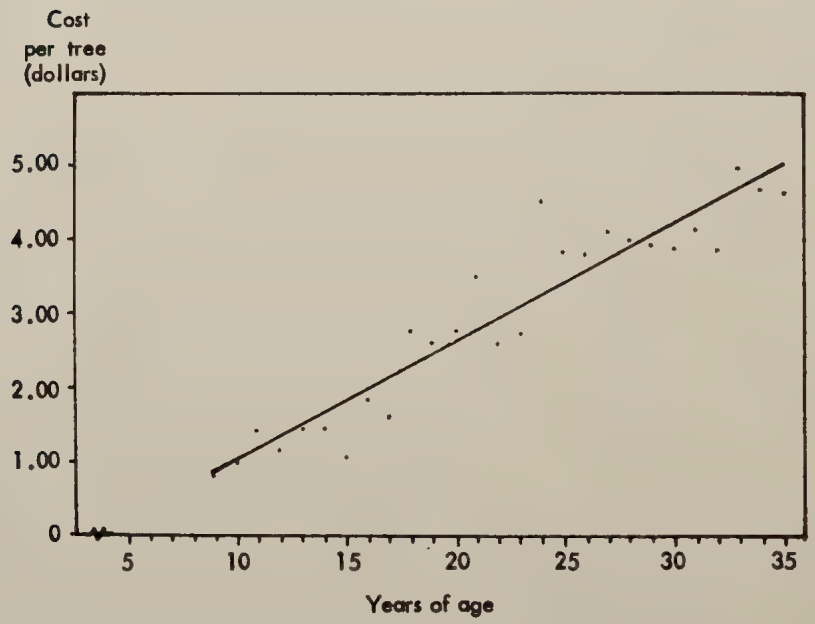


Fig. 119. Relationships between production cost per tree and tree age for Marsh grapefruit grown in southern Lake County, Florida. (From Husmann *et al.*, 1962.)

data were presented for trees only up to 35 years old, some variation might have occurred in the rate at which production declined in later years. However, it can be stated that these trees had reached a stage at which their productive capacity had begun to diminish.

Data from these same plots were used to determine the relationship between the age of the grapefruit trees and the production cost per tree. Figure 119 indicates that this cost increased constantly over the 35-year period, at the rate of about 16 cents per tree per year.

The results of this study seem to indicate that the Florida grower with older grapefruit acreages faces rising costs of production on both per-box and per-tree bases as the trees grow older. Consequently, the cost of production on a per-tree basis increases with tree age, and the cost per box will increase proportionately with declines in yields per tree.

Wood and Reed (1938), in studying the influence of the age of the tree on the maturity of Texas grapefruit, reported that old trees matured their fruit earlier than young ones because higher soluble solids (Brix values) and Brix: acid ratios were present in fruit from the older trees.

Hilgeman (1941), using trees growing in the Salt River Valley of Arizona, presented data from young (7 years) and old (24 years) trees which appeared to reverse the interpretation put on the data reported for Texas. Hilgeman's data strongly indicated that fruit samples from old trees generally had higher Brix values and higher percentages of acid and lower Brix:acid ratios than samples from young trees. In terms of the Brix:acid ratio as an index of commercial maturity, the old trees matured their fruit at a later date than young trees.

Plant Growth Regulators. During the past 3 decades, tests have been made to determine the effects of certain synthetic substances, such as 2,4-dichlorophenoxyacetic acid (2,4-D) and 2,4,5-trichlorophenoxyacetic acid (2,4,5-T), on various fruits while on the trees and after being placed in storage. These and similar substances were formerly called plant "hormones," but now, since they are synthesized outside of the plant, they are called "growth regulators." Their effects have been determined, not only on the fruit but on many of the other tissues of various plants. Our discussion here will be limited to their effects on grapefruit.

Stewart and Parker (1947; 1948*a, b;* 1954) reported results showing that water sprays containing 5 to 25 ppm of 2,4-D caused a marked reduction in fruit drop. The most significant results were obtained in their 1947 tests (see the 1954 citation). Figure 120 clearly illustrates the magnitude of the results obtained by spraying grapefruit with 2,4-D. Other trials showed a reduction of 53 to 65 per cent in fruit drop. In their 1947 tests, the fruit from each treatment was sold separately by the packing house. On a per-tree basis, it was found that the nonsprayed trees grossed $6.40, while those sprayed with 8, 16,

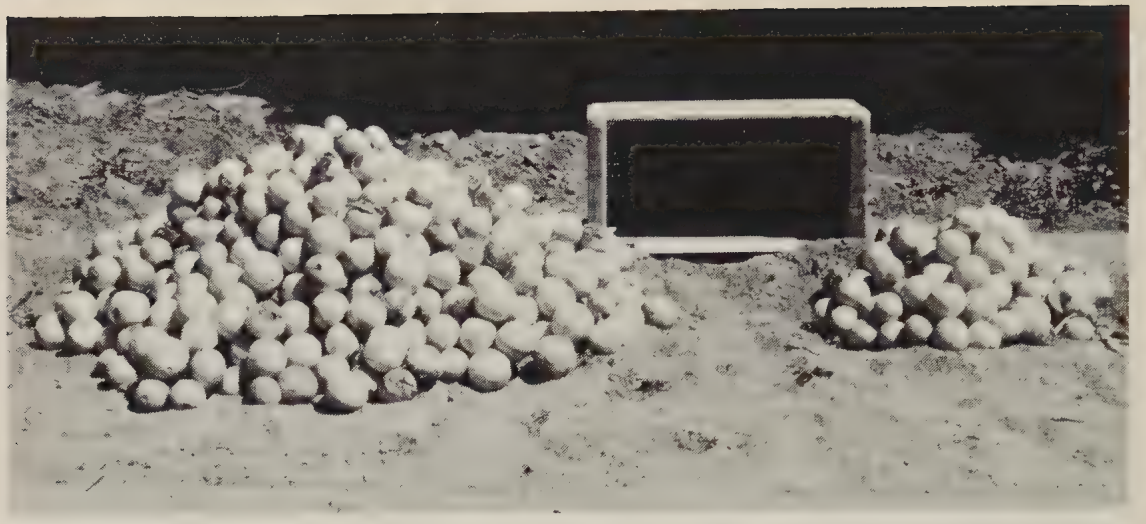


Fig. 120. Mature fruit drop from grapefruit trees at Highgrove, near Riverside, California, June 4 through July 17, 1947. *Left:* 517 fruits from 10 nonsprayed trees. *Right:* 87 fruits from 10 trees sprayed with 8 ppm 2,4-D on June 4. (From Stewart and Parker, 1954. Photo by O. K. Anderson.)

or 24 ppm of 2,4-D yielded $5.34, $8.32, and $10.21, respectively. The increases with 16 and 24 ppm were owing not only to the greater yields, but also to the higher values, at that market, of large-sized fruit. The dates of spray application ranged from April 15 to July, just before harvest. The dates of application and the concentrations of the growth regulator used appeared to have little effect on the composition of the juice.

Stewart (1948, 1949) treated grapefruit with either 8 or 16 ppm of 2,4-D, and placed the fruit under normal storage conditions. At the end of a 15-week storage period, the treated fruit, especially that given 16 ppm, was much superior to the untreated fruit in quality. These values are of interest:

> Nontreated fruit: 88 per cent black buttons; 24 per cent visible decay.
> Treated with 8 ppm: 58 per cent black buttons; 9 per cent visible decay.
> Treated with 16 ppm: 31 per cent black buttons; 5 per cent visible decay.

Stewart also found that 2,4,5-T was more efficient than 2,4-D in reducing black buttons, alternaria rot, and external decay.

Schiffmann-Nadel (1969) found that when grapefruit was treated with a wax coating containing 2,4-D, a higher percentage of fruit retained green stem ends, and lower percentages of stem-end rot and internal core rot occurred during storage. In fruit that had been stored for 12 and 16 weeks at 12° C. (54° F.), the numbers of green stem ends in fruit coated with wax, *without* 2,4-D, amounted to 11.8 and 11.3 per cent, respectively, as compared with 82.8 and 67 per cent for fruit similarly coated with wax *containing* 2,4-D. In a similar storage experiment at 12° C., stem-end rot amounted to 7.4 and 10.8 per cent in grapefruit without 2,4-D, and to 3.4 and 3.5 per cent in fruit treated with 2,4-D. In these experiments, the internal core rot (probably

Alternaria citri) amounted to 2.4 and 3.8 per cent in nontreated fruit as compared with 0 and 1 per cent in fruit treated with 2,4-D. (See also, figure 162 of chapter 5, and accompanying text starting on page 484.)

Cameron (1948) reported that 2,4-D was very effective in reducing winter grapefruit drop near Mildura (Victoria) in Australia. The amount of fruit on the ground under the treated trees was only about 2 per cent of that under the nontreated trees. At the time his paper was written, the optimum concentration of the growth regulator had not been determined.

In Arizona, Hilgeman (1951) sprayed grapefruit trees with 2,4-D, using 8 ppm in 1948, 6 ppm in 1949, and 10 ppm in 1950. The 1950 applications were made to determine the effects of 2,4-D on the drop of frozen fruits. The first one was made on January 17. On April 5, a second application was made on half of the same trees. No significant differences in fruit drop occurred until June, when a heavy drop occurred from the unsprayed trees. In early August, 67 per cent of the fruit had dropped from the control trees, 52 per cent from the trees sprayed in January, and 26 per cent from the trees sprayed in both January and April. These results are shown in the curves of figure 121.

Hilgeman also found that by applying these sprays in April, the time at which the trees were also sprayed for thrips, they effectively prolonged the picking season. The cost of spraying with 2,4-D was practically negligible be-

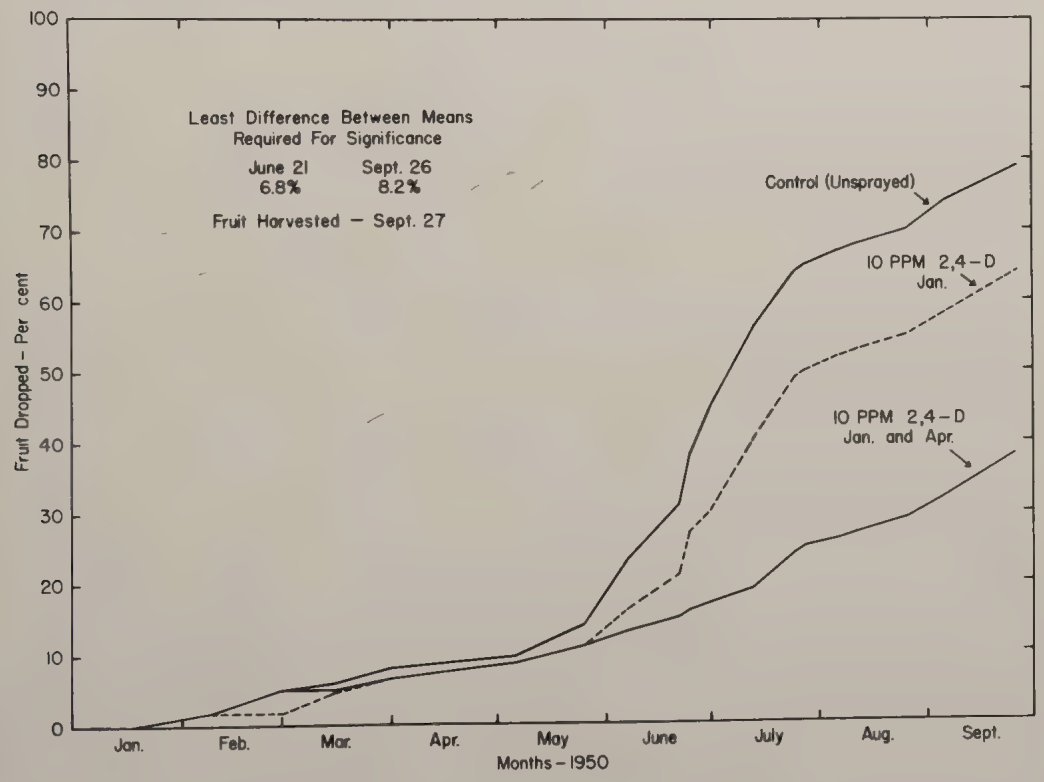


Fig. 121. Effects of 2,4-D spray on the drop of mature grapefruit. (From Hilgeman, 1951.)

cause it could be added to the spray used for controlling thrips. The 2,4-D made the fruit firmer, significantly reduced fruit drop, and did not reduce the quality of the juice.

Erickson *et al.* (1952) pointed out that maleic hydrazide (MH) would act as a plant-growth inhibitor when used at certain concentrations, and as a herbicide when used at higher ones. They applied it at different times of the year, and at concentrations of 100, 500, and 1,000 ppm, to Valencia orange and Marsh grapefruit trees. They found that: (1) in the first new growth after 1,000 ppm had been applied, the leaves were narrow and asymmetrical, 500 ppm had less effect, and 100 ppm had no effect; (2) none of the applications caused any noticeable decrease in fruit drop; however, an increased yield per tree was effected, probably by slightly depressing the natural thinning processes; and (3) no effects were noted on the quality of fruit from the sprayed trees.

Guiscafre-Arrillage (1948) found that the germination of spores of *Penicillium digitatum* (green mold) suspended in 0.1 to 0.3 per cent 2,4-D in different forms was only 14 per cent, as compared with 82 to 91 per cent germination in the checks. This offers an explanation for the reduced amount of decay in stored fruit treated with 2,4-D.

In studying the elongation of citrus shoots as the result of a series of growth cycles, Cooper *et al.* (1964) observed that the terminal growing point of a new shoot was inhibited by the production of new leaves. In the genus *Citrus,* this inhibition causes the terminal growing point to die, which prevents further extension growth of the shoot. It is well known that apical dominance is dependent on the terminal bud, the location in which auxin is synthesized. The growth of lateral buds after removal of the terminal bud is inhibited by the application of auxin to the surface of the decapitated stem.

Cooper (1939) reported that the young, developing leaves of citrus shoots were major sources of auxin, and that they exerted an inhibitory influence on the growth of axillary buds below them. This bud inhibition disappeared as soon as the auxin supply to the buds was cut off by defoliation.

The experiments of Wickson and Thimann (1958) showed that gibberellin and kinin were associated with auxin in inhibiting the growth of lateral buds of the pea plant by apical dominance.

Cooper and Peynado (1958) applied gibberellin to mature, bearing Redblush grapefruit trees during the summer, and found that the buds were released from inhibition similar to defoliation. In some instances, gibberellic acid produced elongation of individual shoots at concentrations of 1,000 ppm, but the total elongation produced by the tree was not increased by the spray treatment. In treated trees, some shoots developed thorns, while others developed long, narrow leaves. The principal physiological characteristic of the gibberellic acid treatment was that of breaking and shortening the dor-

mant period of grapefruit trees that were in states of winter, spring, or summer dormancy. The experiments showed that all buds on a shoot and several growth cycles back on the treated trees broke dormancy, but only a few buds near the apex of the shoot grew into new shoots. The dormant periods of the other buds were broken, but they did not elongate.

In studying the influence of potassium gibberellate on Marsh grapefruit trees and fruit grown under California conditions, Coggins *et al.* (1962) sprayed mature, bearing trees with potassium gibberellate at acid equivalent concentrations of 0, 37.5, 75, 150, and 300 ppm. The trees were sprayed on April 28, 1958, and data were collected from the crop set in 1958 and harvested July 13, 1959. The trees were past full bloom, with small fruits (the 1959 crop), and the mature fruits (the 1958 crop) were completely yellow in color at the time of treatment. The spray treatment caused severe leaf drop and twig dieback, with regreening of the 1958 crop and loss of fruit turgor. The 1959 crop that came from trees treated in 1958 showed many fruits with incomplete loss of chlorophyll from the rind, resulting in undesirable color with a corresponding reduction in crop value. It would be difficult to delay softening and aging of grapefruit rind tissue without the occurrence of considerable regreening of the fruit. Gibberellic acid decreased the drop of mature fruit, but reduced the production on treated trees. (See also, Coggins and Lewis, 1962.)

In other experiments, Coggins *et al.* (1966) noted that Redblush grapefruit dipped in 500 ppm of gibberellic acid developed a green ring on the stylar end within 4 months after treatment, and that brown specks persisted on the treated fruits until harvest.

Lewis *et al.* (1967), in reporting on experimental data limited to the navel orange, described some interesting results which showed that biochemical changes were associated with gibberellin A_3 in delaying senescence in the rind. The total and reducing sugars in the albedo of the treated and control fruit accumulated to approximately the same concentrations up to March 4; from then on, the concentrations of soluble sugars in the treated fruit fell behind those of the controls. The sugar accumulation in the rind was highly dependent on the relationship between the quantity transported to the albedo and the amount respired. The reduced rate of accumulation of soluble sugars by the treated fruit was highly associated with the corresponding increase in the rate of respiration based on oxygen absorption. The treated fruit had a greater net respiration rate as well as a greater rate of glucose utilization. An analysis of the data seems to indicate that the treated fruit had a higher metabolic rate than the control fruit; consequently, the treated fruit had a slower rate of accumulation of soluble sugars late in the season. The authors emphasized that these seasonal differences between fruit treated with gibberellic acid and control fruit occurred around March. At this period, the

treated fruit was observed to have higher rates of oxygen uptake, higher rates of glucose respiration, a reduced amount of soluble sugars, a lower K/Ca + Mg ratio, and a higher level of phosphorus. March was the period which marked the end of carotenoid accumulation, the beginning of the accumulation of rind exudate, and the stage at which gibberellic acid would no longer accelerate rind firmness.

Abscission of Citrus Fruit in Relation to Ethylene and Ethylene-Inducing Compounds. All ripening fruits have been shown to emanate ethylene in various amounts under different environmental conditions. As discussed in chapter 5, those fruits that demonstrate a postharvest climacteric increase ethylene production concomitantly with increased respiration during the climacteric rise. Both ethylene formation and respiration decrease with senescence. During ripening, citrus fruits behave differently in that there occurs no marked upsurge in respiration rate with the initiation of color in the rind. In citrus fruit pulp, there is no reserve carbohydrate, such as starch, which can be converted into soluble sugars and subsequently serve as a source of energy for respiration, as there is in those fruits which exhibit a typical climacteric rise in respiration during the ripening period.

It has been shown that NAA (1-naphthaleneacetic acid), 2,4-D (2,4-dichlorophenoxyacetic acid), 2,4,5-T (2,4,5-trichlorophenoxyacetic acid), and other related compounds not only prevent fruit abscission, but they also have been reported to induce ethylene in plants (Cooper and Henry, 1967; Palmer *et al.*, 1967; Rasmussen and Cooper, 1968). Since ethylene is highly efficient in accelerating abscission, and since synthetic auxins induce ethylene in plants when applied exogenously, it follows that auxin should accelerate abscission. This line of reasoning has led to an area of research which involves screening tests for compounds that induce ethylene in citrus and other fruits without possessing fruit-abscission properties.

In their review on the physiological basis of abscission in agricultural crops, Cooper *et al.* (1968) emphasized the nature of the dual problem of fruit abscission, which includes both the prevention of preharvest drop and the loosening of fruit at harvest time. During the last 35 years, auxin-containing sprays, such as 1-naphthaleneacetic acid (NAA) and 2,4-dichlorophenoxyacetic acid (2,4-D), have been applied to fruit trees to prevent the premature drop of apples, pears, peaches, and citrus fruits.

Gardner *et al.* (1939), following the report of LaRue (1936) which showed that indoleacetic acid delayed the abscission of leaf petioles of *Coleus* plants, experimentally determined that the preharvest drop of apples could be significantly delayed by spraying the foliage and fruit with synthetic auxins, such as naphthaleneacetic acid and naphthaleneacetamide.

The acceleration of ripening and subsequent senescent changes that occur in many fruits, caused by ethylene gas, have been amply demonstrated. The

precise role of ethylene in producing the climacteric rise in respiration has not been clearly defined. Several investigators (Burg and Burg, 1965; Meigh *et al.*, 1967; Von Abrams and Pratt, 1967) maintained that ethylene initiated the climacteric, but Wilkinson (1963) suggested that ethylene production was a result of biochemical and physiological changes that occurred in the preclimacteric and climacteric phases of fruit ripening. In other words, ethylene might be a by-product and not an initiator of the ripening process. Considerable evidence has been advanced to support the idea that ethylene contributes to the over-all ripening phase of fruits by causing membrane changes, resulting in increased respiration and greater activity of certain membrane-oriented enzymes (Sachar, 1962; Looney and Patterson, 1967). Indole-3-acetic acid (IAA) was shown to stimulate ethylene evolution in various plant tissues and fruits (Abeles, 1966; Burg and Burg, 1966). Since gibberellins are not associated with ethylene production in citrus fruits, they were found to have opposite effects on ripening and senescence (Dostal and Leopold, 1967; Scott and Leopold, 1967). According to Cooper *et al.* (1968), gibberellin A_3 produced abscission activity in the explant assay, produced ethylene when applied to citrus leaves, but did not produce it when applied to citrus fruits.

During the past several years, many chemical compounds have been applied, in dilute solutions, as sprays to citrus trees to induce loosening of the fruit. The abscission of citrus fruits has taken on a considerable degree of importance as a result of the development and application of mechanical harvesting methods to the trees.

Hendershott (1964, 1965) reported that of the 27 chemical compounds that were screened, iodoacetic acid (Na salt) proved to have the greatest effect on citrus fruit abscission. The degree of loosening of the fruit by iodoacetic acid was measured by determining the force (in pounds), on a straight pull, required to remove the stem from the fruit. The data illustrated in figure 122, regardless of any commercial significance, indicate the effects of different concentrations of iodoacetic acid on the relative looseness of fruit, as detected on samples at various intervals for 8 days after spraying. The degree of force required to loosen the fruit was noticeably reduced 2 days after spraying, and the maximum loosening was observed between the third and sixth days.

Hendershott's research initiated interest in the problem of citrus fruit abscission. Cooper and Henry (1967) noted that growth-regulator effects on the abscission of bean-leaf petiole explants might not be highly correlated with the abscission of citrus fruits. Further research may reveal that abscission of both leaves and fruit is caused by the same physiological mechanism.

Scott *et al.* (1948) arrived at the following conclusions: Citrus leaves are similar to compound leaves in that the lamina, which may be considered a terminal leaflet, frequently abscises before the petiole. Both laminar and nodal abscission zones are structurally weak in unlignified pericyclic fibers,

and have a low content or complete lack of xylem fibers. The structural changes that are highly associated with leaf fall occur in the parenchymatous cells of the abscission zone. The physiological and anatomical changes in both the laminar and nodal abscission zones are similar, in that (a) starch disappears with partial replacement by calcium oxalate; (b) there is suberization of the basal tissues of the lamina; (c) differential growth of the abscission parenchyma tissue occurs; and (d) finally, there is a break of tissue at the separation layer. The laminar abscission region has a definite abscission zone which is composed of a band of cells about 10 layers deep, lying at the distal

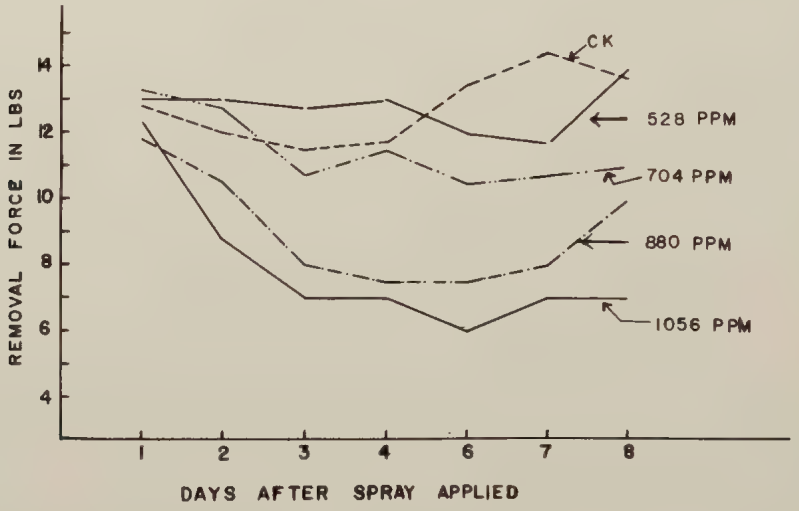


Fig. 122. The effects of iodoacetic acid (sodium salt) on abscission induction in fruit of the Pineapple orange. (From Hendershott, 1965.)

end of the petiole almost level with the groove, and distinguished by smaller cell dimensions. Abscission eventually occurs along the separation layer—the distal face of the abscission zone. The nodal abscission region has no definitive abscission tissue. The break is first indicated in mature leaves by differential distribution of starch and intercellular spaces.

Cooper *et al.* (1968), when conducting field experiments in Florida, determined the effects of various growth regulators on preventing the abscission of Hamlin oranges. Their experimental data are reported in table 163 in terms of the pull force required to remove the stem from the fruit, the tearing of the rind on picking, the cumulative fruit drop per tree, and the effect on color development of the fruit. The spraying of 2,4,5-T and gibberellin A_3, respectively, on Hamlin orange trees during the 1967 and 1968 seasons produced a substantial reduction in fruit drop. The gibberellin A_3 treatment for the 1967 season produced a tightening of the fruit instead of loosening it. In fact, some regreening occurred in the oranges, while the 2,4,5-T treatment increased the orange color of the rind.

TABLE 163

THE EFFECTS OF VARIOUS GROWTH REGULATORS ON PREVENTING FRUIT ABSCISSION OF HAMLIN ORANGES IN FLORIDA (1967–68).*
(Tests were conducted at the U. S. Horticultural Field Station, Orlando, Florida.)

Materials applied 11/22†	Concentrations (micromoles)‡	Pull force (kilograms)			Tearing of rind on picking (per cent)			Cumulative fruit drop per tree			Fruit colors on 3/1§
		11/29	1/11	3/1	11/29	1/11	3/1	11/29	1/11	3/1	
Water (control)	...	6.3	6.3	3.9	90	30	00	5	30	176	J
2,4,5-T	150	6.8	6.3	5.0	80	100	50	3	8	40	K
2,4,5-T	10	6.3	6.3	4.1	100	100	20	0	11	57	J
GA_3	280	6.3	7.7	5.9	100	100	40	1	13	44	I
GA_3	70	7.2	7.7	4.6	100	100	20	1	13	91	I
BA	440	6.3	6.8	3.6	100	100	00	4	10	141	J
BA	110	6.8	6.3	3.9	100	90	00	7	20	230	J
B-995	3,100	7.2	6.8	3.6	90	90	00	20	32	174	J

*From Cooper *et al.* (1968).
†2,4,5-T is 2,4,5-trichlorophenoxyacetic acid; GA_3 is gibberellic acid (K salt); BA is benzyladenine; B-995 is N-dimethylaminosuccinamic acid.
‡Averages for 10 fruits tested on the indicated dates.
§Color ratings were made according to the color chart of Harding *et al.* (1940) for oranges. A rating of K is more highly colored than J, and I is less highly colored. There was a trace of regreening at the stem end of GA_3-treated fruit.

Lewis and Bakhshi (1968) observed that GA_3 accelerated the abscission of citrus leaves, but not of the fruit. Coggins and Lewis (1962) had reported earlier that fruit from Valencia orange trees sprayed with GA_3 regreened and developed the peel characteristics of juvenile fruit.

Rasmussen and Cooper (1968) reported on the stimulation of ethylene production by various chemical substances applied exogenously to the fruits of the calamondin (*Citrus mitis*). Their data shown in table 164 were obtained from ripe, detached fruits which had been dipped into the chemicals for 1 minute, dried, placed in quart jars, and sealed by rubber stoppers with plastic tube connections. Ethylene was determined on aliquot samples taken by syringe through the plastic tubes at 18 and 42 hours after treatment. Ascorbic acid at 1 per cent concentration stimulated ethylene production, but at 0.5 per cent concentration, the ethylene produced was not significantly different from that of untreated fruit. FeEDTA (ferric ethylenediaminetetracetic acid)

TABLE 164

ETHYLENE EVOLUTION FROM CALAMONDIN FRUITS DIPPED INTO SOLUTIONS OF ASCORBIC ACID, COMBINATIONS OF ASCORBIC ACID WITH FeEDTA AND CuEDTA, AND SOLUTIONS OF FeEDTA AND CuEDTA.*

Treatments	Ethylene (ppm)	
	18 hours	42 hours
1 per cent AA	0.67	0.85
1 per cent AA + 0.1 per cent FeEDTA	.83	1.32
1 per cent AA + 0.05 per cent FeEDTA	.63	.68
1 per cent AA + 0.01 per cent FeEDTA	.60	.59
0.5 per cent AA	.30	.32
0.5 per cent AA + 0.1 per cent FeEDTA	.63	1.00
0.5 per cent AA + 0.05 per cent FeEDTA	.49	.82
0.5 per cent AA + 0.01 per cent FeEDTA	.32	.37
0.1 per cent FeEDTA	.53	.68
0.01 per cent FeEDTA	.32	.35
1 per cent AA + 0.1 per cent CuEDTA	1.87	2.68
1 per cent AA + 0.05 per cent CuEDTA	1.53	2.45
1 per cent AA + 0.01 per cent CuEDTA	.71	.84
0.5 per cent AA + 0.10 per cent CuEDTA	1.67	2.74
0.5 per cent AA + 0.05 per cent CuEDTA	1.21	2.38
0.5 per cent AA + 0.01 per cent CuEDTA	.33	.53
0.1 per cent CuEDTA	1.57	2.88
0.05 per cent CuEDTA	.55	.67
0.01 per cent CuEDTA	.26	.34
Untreated	0.22	0.27

*From Rasmussen and Cooper (1968).

treatment at 0.1 per cent concentration increased ethylene production, in combination with ascorbic acid or alone, with the exception of 0.05 per cent FeEDTA plus 0.5 per cent ascorbic acid, which was almost equivalent to the 1 per cent ascorbic acid treatment after 42 hours. CuEDTA (cupric ethylenediaminetetracetic acid) was superior to FeEDTA, alone or in combination with 1 or 0.5 per cent ascorbic acid, in accelerating ethylene production by calamondin fruit. At concentration levels of 0.1 and 0.05 per cent, CuEDTA plus 1 per cent ascorbic acid produced ethylene at a rate 3 times that of the FeEDTA treatments. CuEDTA did not produce ethylene significantly at concentrations of 0.01 per cent, alone or in combination with 0.5 per cent ascorbic acid. The combination of ascorbic acid with 0.1 per cent CuEDTA stimulated ethylene at approximately the same rate as 0.1 per cent CuEDTA alone.

Rasmussen and Cooper (1968) also sprayed calamondin seedlings with various concentrations and combinations of chemicals to ascertain the relative degrees of fruit abscission under specified conditions. Half of the treated seedlings were placed in 18-liter (roughly, 5-gallon) glass jars in which the evolved ethylene was trapped and measured 18 hours after the treatment; the other half were placed in a greenhouse, where abscission counts could be made 4 days after the treatment. Fruit abscission was stimulated by the following spray applications: 2 per cent AA (ascorbic acid); 1 per cent AA plus 0.05 or 0.10 per cent CuEDTA; 1 per cent AA plus 0.1 per cent FeEDTA; and 0.5 per cent AA plus 0.10 per cent CuEDTA. The air around the seedlings in the jars had an ethylene content ranging from 0.14 to 1.20 ppm. These investigators accordingly concluded that all chemicals and combinations of chemicals tested that induced abscission of citrus fruit also accelerated ethylene production by calamondin fruits and seedlings. Conversely, not all the chemical substances that induced ethylene production necessarily induced abscission of citrus fruit, because of uncontrollable variables.

Palmer *et al.* (1969), in recognition of the importance of ethylene in the abscission of both citrus leaves and fruits, showed the relationship between the force of attachment and the ethylene evolution from stem and abscission zone tissues (figure 123). The experimental materials consisted of abscission zone tissue and stem tissue 1 centimeter in length adjacent to the abscission zone. Detached navel orange fruit gassed with 20 ppm of ethylene abscised within 48 hours, with a 70 per cent reduction of the initial pull force, without increasing ethylene biogenesis in the cells of the abscission plug. The increased rate of abscission from the ascorbic acid treatments was associated not only with increased yields of ethylene, but also with reduction of the attachment force of the fruit stem. The interaction between these variables was highly significant. The detached fruit dipped in 5 per cent ascorbic acid produced 50 per cent reduction in pull force within 48 hours. This definite reduction in pull force was accompanied by an acceleration in ethylene produc-

tion 10 times that of the ethylene-treated fruit. Ethylene yields continued to increase with further reduction in pull force.

In a manner similar to the 5 per cent dip treatment of detached fruit, the 2 per cent ascorbic acid solution that entered the stem stimulated ethylene production in the abscission zone plug (figure 123). Similar feeding experiments with lower concentrations of ascorbic acid (0.5 and 1 per cent) accelerated abscission, but at a reduced rate. The treatments of abscission zone plugs from fruit with either 1 or 2 per cent ascorbic acid were identical in their interactions between loosening of the fruit and ethylene production.

Palmer *et al.* (1969) also reported on the effects of 2,4-D on ethylene production, and the reduction in pull force of early-ripe navel oranges as an indicator of abscission. A 50 per cent reduction in pull force occurred in the control fruit before ethylene increased beyond the initial rate of 7 milli-

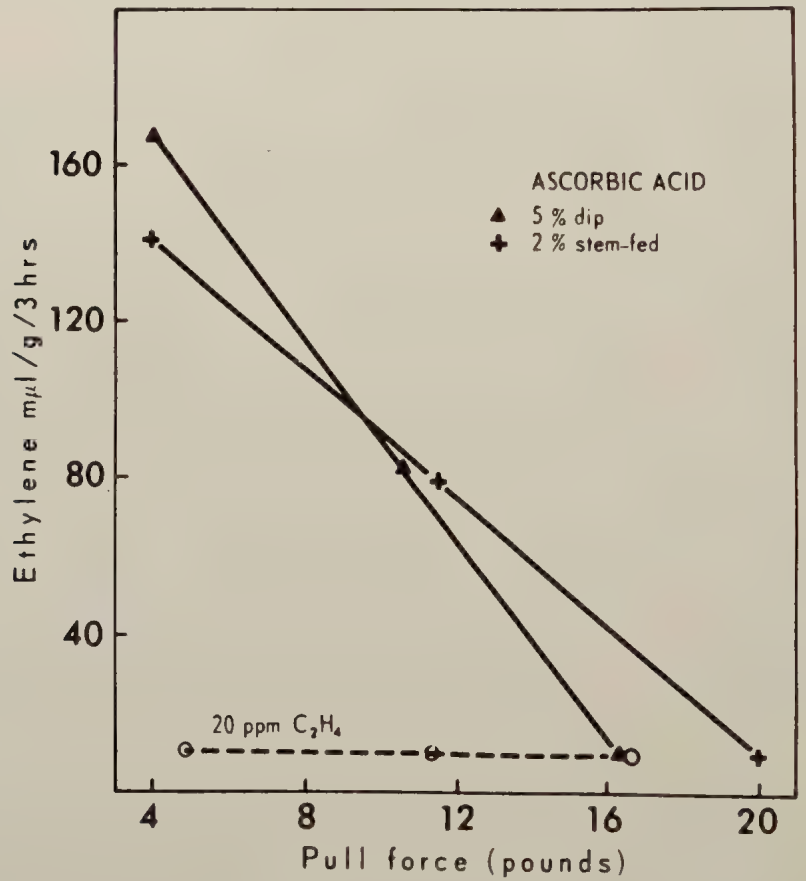


Fig. 123. Reduction in pull force and associated ethylene production by tissue plugs containing the abscission zone of midseason ripe navel orange fruit treated with ascorbic acid or ethylene gas. Calculated *t* (tests for significance) values for the 5 per cent dip and 2 per cent stem-fed treatments were 2.56 and 2.53, with 99 and 39 degrees of freedom, respectively. (From Palmer *et al.*, 1969.)

microliters per gram in 3 hours (table 165). In the control fruit, ethylene production finally reached a rate of 300 millimicroliters per gram in 3 hours from the abscission zone tissue, accompanied by a 95 per cent reduction in pull force. The stem tissue significantly accelerated ethylene emanations from 12 to 320 millimicroliters per gram in 3 hours from the sixth to the seventh day. The auxin 2,4-D accelerated the ethylene production by the abscission zone at a significantly higher rate than the controls up to the sixth day. The 2,4-D inhibited abscission, and the rate of ethylene emanations did not increase after the seventh day. Ethylene production increased from the stem at the seventh day, and this acceleration appeared to be related to similar senescence activity observed in the controls. It was suggested that 2,4-D prevented degradation of the abscission zone by the endogenous ethylene by accelerating metabolic reactions which not only increased ethylene yields, but prevented abscission.

Palmer *et al.* (1967) reported experimental evidence which indicated that ethylene produced from *beta*-hydroxyethylhydrazine (BOH) accelerated the abscission of detached fruits and leaves in closed containers. Experiments were performed on detached citrus fruits with 2-inch (5-centimeter) stems which were placed in a desiccator with their stems immersed in water, or placed individually in test tubes and enclosed in polyethylene bags. Aqueous solutions of BOH ranging from 50 to 1,000 ppm induced abscission in oranges, grapefruit, and lemons. The rate of abscission of the treated fruit is well illustrated in figure 124 by the rapid reduction in pull force needed to

TABLE 165

EFFECTS OF 2,4-D ON ETHYLENE PRODUCTION AND REDUCTION IN PULL FORCE OF EARLY-RIPE NAVEL ORANGES.*

Factors	Number of days						
	1	2	4	6	7	8	9
Abscission zone:							
+2,4-D	20[b]†	34[b]	25[b]	15[a]	43[a]	45[b]	43[b]
−2,4-D	7	8	7	6	66	110	300
Stem:							
+2,4-D	27[b]	110[b]	81[b]	80[b]	900[b]	680[b]	500[b]
−2,4-D	7	8	6	12	320	330	260
Controls:							
Percentage of reduction in pull force	0	0	0	25	50	65	95

*Stated as millimicroliters of ethylene per gram produced in 3 hours. (From Palmer *et al.*, 1969).
†The rates of ethylene synthesis were significantly different from the controls (−2,4-D) of the same age at the 5 per cent confidence level (a) or the 1 per cent level (b).

separate the stem from the fruit. The pull force, as a measure of abscission, was reduced about 50 per cent within 15 hours.

These researchers also noted that abscission induced by BOH was caused by the chemical conversion of this compound into ethylene. This statement was substantially supported by data from experiments which consisted of placing detached citrus fruits in closed containers with their stems in water

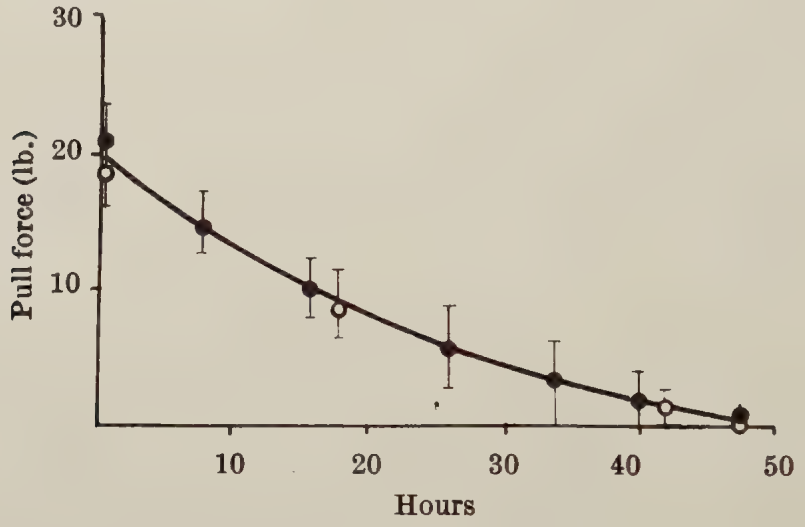


Fig. 124. Reduction in pull force needed to separate Washington Navel fruit from the stem, using 100 ppm *beta*-hydroxyethylhydrazine in a closed chamber. (From Palmer *et al.*, 1967.)

and adjacent to an open beaker containing 1,000 ppm of aqueous BOH. The fruit in these containers abscised in approximately 60 hours, which indicated that the substance inducing fruit abscission had to be liberated from the aqueous BOH in gaseous form. This gaseous substance, based on analyses of air samples from the containers, proved to be ethylene. The data support the view that the abscission of citrus fruit explants induced by *beta*-hydroxyethylhydrazine is highly associated with the ethylene produced as a result of the decomposition of the BOH molecule.

Maturity Standards

THE FIRST ATTEMPT to establish maturity standards with legal backing occurred in the Florida Legislature over 40 years ago. Since that time, many states and countries have passed laws concerning and defining maturity standards for different kinds of citrus fruits (Taylor, 1940).

Criteria. No single standard thus far used has proved entirely satisfactory. Some of the difficulties encountered in using various criteria of maturity are given on the following page.

Color of Fruit. One fruit may be only 20 to 25 per cent colored, while another with the same degree of palatability may be 70 per cent colored.

Peel Percentage or Thickness. The wide range in percentages of peel and peel thickness in fruit with the same degree of palatability makes these factors meaningless as maturity measurements.

Total Soluble Solids. Seasonal changes in the percentage of total soluble solids in the juice are comparatively slight. These values alone are meaningless for determining fruit maturity. Hilgeman (1941) reported that *immature* fruit from one grove contained 12.89 per cent total soluble solids in September, while *mature* fruit from another grove contained only 10.17 per cent in December.

Total Acids. Hilgeman reported further that a high percentage of total soluble solids was usually accompanied by a high percentage of acids, and a low percentage of total soluble solids by a low percentage of acids. For this reason, and because fruit with a low percentage of acid is palatable with a lower percentage of total soluble solids than more acid fruit, a definite relation between percentages of acids and palatability is often difficult to establish.

Solids:Acid Ratio. Heat summation and other environmental factors, as well as irrigation and other cultural factors, may affect both total soluble solids and total acids, but not necessarily to the same relative degree. Thus, in California, fruit from districts with high heat summation usually has not only higher percentages of total solids and acids but also a higher solids:acid ratio than that from cooler districts. These facts are taken into consideration in the California legal standards for grapefruit maturity, a higher solids:acid ratio being required from districts with higher heat summation. In Florida and Texas, the required ratio varies inversely with the total soluble-solids content. Furthermore, the accuracy of the ratio determinations is affected by a number of factors, such as the type of juice extractor used, the amount of air incorporated during extraction, and the representativeness of the sample.

Because of juice gradients in solids and acid content (see, for example, table 49 of chapter 2), it is important to obtain complete extraction of all the juice. The position of the sampled fruit on the tree is important, for fruits from the south and west sides of the tree usually have a higher solids:acid ratio than those from the east and north sides. The ratio also depends on the operator; the same lot of juice may yield different results when tested by different operators or by the same operator at different times.

Harding (1947*b*) distinguished the terms "mature" and "ripe" by stating that "mature" refers to the stage of development of the fruit, while "ripe" refers to the processes by means of which a mature fruit becomes edible when held under proper conditions. Since with grapefruit the ripening processes occur only while the fruit is on the tree, it can be readily understood that it should not be harvested until it is mature, and therefore ripe. Instead of increasing in good quality after harvest, the fruit tends to lose it. The rate of loss depends on the temperature at which it is held: the higher the temperature, the more rapid the deterioration. A wholly green fruit may be fully ripe under certain natural conditions, while a fully yellowed fruit may be immature under other conditions. It is the composition of the fruit and not its color that determines ripeness. It is as ripe as it ever will be when it is picked.

Baier (1932) made extensive maturity tests on grapefruit during the period from 1922 to 1931 from 4 areas in California and 2 in Arizona. Tests were made on (1) ratio, (2) color, (3) tenderness of pulp, (4) amount of juice, (5) bitterness, (6) astringency, (7) different kinds of sugars, (8) ash, and (9) amino nitrogen. From comparison of these with taste tests, he concluded that if the results of the first 4 were satisfactory, the 5 remaining factors would not have to be considered in determining maturity. He made the general statement that the minimum standard of maturity would be reached when the pulp was tender enough to be eaten with a spoon, and tart in flavor, without any unpleasant degree of sourness, bitterness, or astringency. He stated that taste (or rather, flavor, which is a combination of taste plus aroma) would have to be given fundamental consideration in determining maturity.

Longfield-Smith (1935*b*) investigated the possibility of substituting pH for acid titration to determine the maturity of the fruit. There was a general correlation between pH and maturity, but it was not consistent enough for field use. He stated that one of the most important tests for the maturity of grapefruit involved its juice content. His findings on the effects of different methods of squeezing on the amounts of juice obtained were discussed earlier (Longfield-Smith, 1935*c*, page 354).

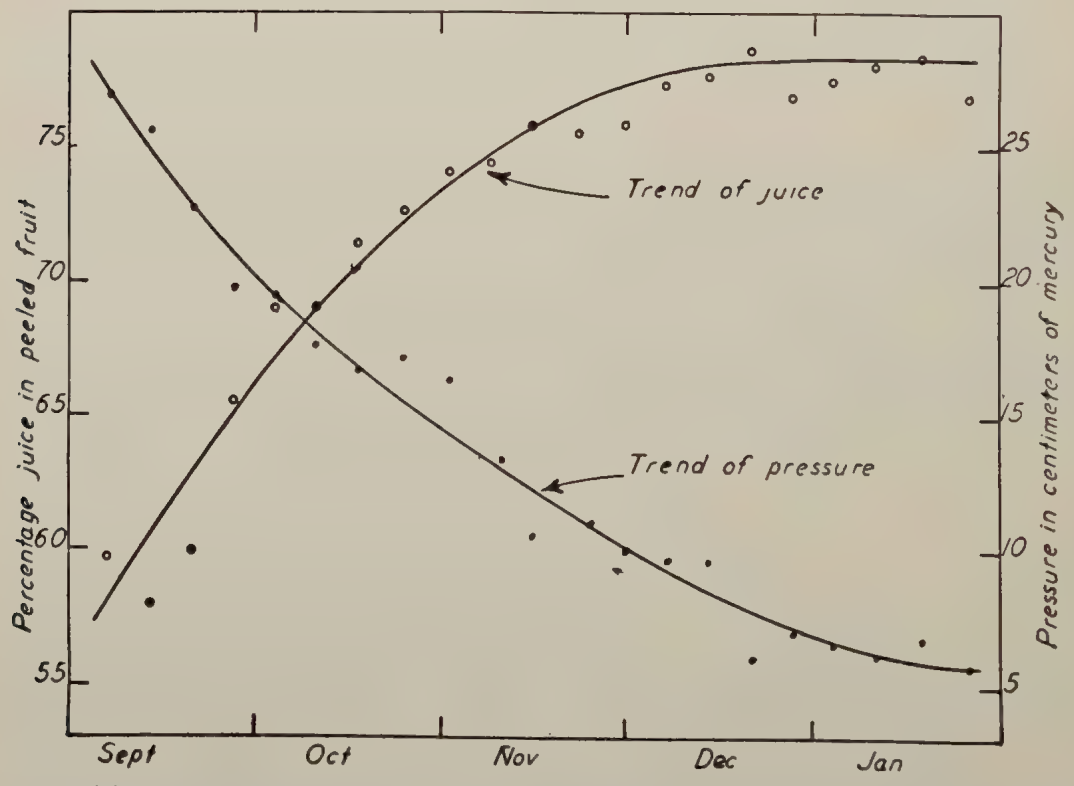


Fig. 125. Seasonal changes in the percentages of juice in the edible portion, and in the pressure values (tenderness) of Marsh grapefruit. (From Hilgeman, 1943.)

Joachim and Panditteseker (1939) stated that maturity standards varied because the solids:acids ratio for good quality varied in different areas.

In Arizona, Hilgeman (1941), as well as others, found that small grapefruit had a higher percentage of juice, by volume and by weight, than large fruit. The difference by weight was found to be less than the difference by volume because the peel had a lower specific gravity than the edible portion. The legal standards for maturity take this difference into account. Under these conditions, the large fruits have a higher solids:acids ratio than the small ones. Hilgeman's data were based on 328 tests made during 5 years.

Hilgeman (1943) reported the results of an attempt to measure maturity in Marsh grapefruit by tenderness tests. He found a positive correlation between tenderness of the flesh and juiciness, but concluded that to adapt such a test to legal maturity standards would require the development of a special type of pressure tester. For the relationship between pressure and juiciness of fruit, see figure 125.

Seltzer (1950), in Arizona, stated that the contemporary standards concerning taste tests for maturity and ripeness (solids:acids ratio) failed to reflect the actual taste, and that a trained taste panel would be more suitable.

Rose *et al.* (1951) pointed out that grapefruit must be mature and of good eating quality when they are picked. When harvesting should be done depends on the chemical and physical condition of the fruit. If they are not "good to eat," the mere fact that they pass some chemical tests will not make consumers think they are ripe.

Krezdorn and Cain (1952), in Texas, stated that in most citrus-growing areas, maturity was based mainly on (1) Brix (total soluble solids, mostly sugars); (2) total titratable acids (mostly citric); (3) solids:acids ratio; and (4) percentage of juice content. Other factors named were esters, organic acids, and glycosides.

The commercial production of grapefruit in the United States is confined almost entirely to 4 states: Florida, Texas, Arizona, and California. The legal maturity standards for these states follow.

Florida. One of the methods for determining maturity of grapefruit in Florida is the use of a "sliding scale" of soluble solids to acids, as shown in table 166. In table 167 are shown the amounts of juice that fruits of different sizes must contain in order to be considered mature. A third maturity standard states that natural yellow color must predominate on not less than 25 per cent of the fruit surface in the aggregate. See the Florida Citrus Code for 1963 (Florida Department of Agriculture, 1963) for other maturity standards relating to fruit that is to be used for processing.

Texas. Section 3. That within the purpose and meaning of this Act, pomelos (grapefruit) shall be deemed to be mature only when the ratio of total soluble solids of the juice thereof to anhydrous citric acid is as follows:

(a) When the total soluble solids of the juice is not less than nine per cent (9%), the minimum ratio of total soluble solids to the anhydrous citric acid shall be seven and two-tenths to one (7.2:1).

(b) When the total soluble solids of the juice is not less than ten per cent (10%), the minimum ratio of total soluble solids to the anhydrous citric acid shall be seven to one (7:1).

(c) When the total soluble solids of the juice is not less than eleven per cent (11%), the minimum ratio of total soluble solids to the anhydrous citric acid shall be six and eight-tenths to one (6.8:1).

(d) When the total soluble solids of the juice is not less than eleven and one-half per cent (11.5%), the minimum ratio of total soluble solids to the anhydrous citric acid shall be six and one-half to one (6.5:1).

Arizona. The Arizona law states that grapefruit are mature if, at the time of picking and at all times thereafter: (1) the juice contains soluble solids, as determined by a Brix scale hydrometer, of not less than 6 parts to every part of acid contained in the juice, the acidity of the juice to be calculated as citric acid without water of crystallization; and (2) not less than 90 per cent of the grapefruit, by count, have attained on at least two-thirds of the fruit surface a minimum characteristic yellow or grapefruit yellow, as indicated by color plate number 19-L3 in Maerz and Paul's *Dictionary of Color.*

California. Grapefruit are not mature unless (a) at the time of picking and at all times thereafter, the juice contains soluble solids, as determined by a Brix scale hydrometer, equal to or in excess of 5 and one-half parts to every part of acid contained in the juice (the acidity of the juice to be calculated as citric acid without water of crystallization), except that in view of differences in climatic conditions prevailing south and east of San Gorgonio Pass which result in the grapefruit grown in that area having, at maturity, a higher percentage of soluble solids to acid than the mature grapefruit grown in the area north and west of said San Gorgonio Pass, grapefruit produced in the area south and east of San Gorgonio Pass are considered mature if at the time of picking and at all times thereafter the juice contains soluble solids, as determined by a Brix scale hydrometer, equal to or in excess of 6 parts to every part of acid contained in the juice (the acidity of the juice to be calculated as citric acid without water of crystallization); and (b) 90 per cent or more of the grapefruit, by count, at time of picking and at all times thereafter have attained, on at least two-thirds of the fruit surface, at least a minimum characteristic yellow or grapefruit color, as indicated by color plate number 19-L3 in *Dictionary of Color,* Maerz and Paul, first edition, 1930. Grapefruit produced outside of this State under climatic conditions similar to those prevailing in the area south and east of San Gorgonio Pass and offered for sale in this State shall meet the same maturity standard as that prescribed for grapefruit produced south and east of said San Gorgonio Pass.

Owing to climatic and other conditions, grapefruit produced in this State south and east of San Gorgonio Pass vary in quality and other particulars from grapefruit produced elsewhere in the State.

The area south and east from San Gorgonio Pass to the north end of the Salton Sea and known as Coachella Valley is hereby designated as the Coachella Valley citrus district. The area comprising Imperial County is hereby designated as the Imperial citrus district. Grapefruit produced in any citrus district may be labeled with the name of the citrus district in which produced. It is unlawful to label any grapefruit with the name of any citrus district unless the grapefruit was produced in such citrus district.

The fruit must not only meet the demands of maturity laws, but also the requirements concerning the pathological and physical condition of the fruit. It must also conform with the provisions of the Federal Food, Drug, and Cosmetic Act.

TABLE 166

MINIMUM RATIOS OF TOTAL SOLUBLE SOLIDS OF GRAPEFRUIT JUICE TO ANHYDROUS CITRIC ACID.*

Percentages of soluble solids		Ratios of soluble solids to acids (minimum)	Percentages of soluble solids		Ratios of soluble solids to acids (minimum)
Minimum	Maximum		Minimum	Maximum	
6.5	9.1	7.1	10.6	10.7	6.35
9.1	9.2	6.95	10.7	10.8	6.325
9.2	9.3	6.90	10.8	10.9	6.30
9.3	9.4	6.85	10.9	11.0	6.275
9.4	9.5	6.80	11.0	11.1	6.25
9.5	9.6	6.75	11.1	11.2	6.225
9.6	9.7	6.70	11.2	11.3	6.20
9.7	9.8	6.65	11.3	11.4	6.175
9.8	9.9	6.60	11.4	11.5	6.15
9.9	10.0	6.55	11.5	11.6	6.125
10.0	10.1	6.50	11.6	11.7	6.10
10.1	10.2	6.475	11.7	11.8	6.075
10.2	10.3	6.45	11.8	11.9	6.05
10.3	10.4	6.425	11.9	12.0	6.025
10.4	10.5	6.40	12.0	12.0	6.00
10.5	10.6	6.375			

*Adapted from the Florida Citrus Code of 1963.

TABLE 167

THE MINIMUM JUICE CONTENTS PERMISSIBLE IN FRUITS OF DIFFERENT SIZES AT DIFFERENT DATES.*

Sizes of fruit (number per packed box)	Minimum juice contents at various dates (milliliters per fruit)		
	August 1 to October 15	October 16 to March 1	March 2 to July 31
28	400	380	360
36	350	335	320
46	320	305	290
54	280	270	255
64	255	240	230
70	230	220	210
80	210	200	190
96	185	180	170
112	175	170	160
126	165	160	150
150	145	135	130

*Adapted from the Florida Citrus Code of 1963.

Foreign Countries. The following methods for determining maturity of grapefruit have been reported through personal correspondence with the authorities in charge of agriculture or horticulture in the foreign regions listed. These maturity requirements and the methods for their determination were in effect in 1965.

Algeria. Use solids:acid test. No values given.

Argentina (Buenos Aires). No maturity standards are enforced.

Australia (Brisbane). Fruit to be clean, well grown, and free from blemish, with smooth skin of a clear yellow color and diameter not less than 3 and one-half inches (9 cm). The peel thickness must not exceed one-fourth inch (about 6.5 mm).

Brazil. Fruit must contain 40 per cent juice by weight; ratio, 6.5:1.

British Guiana. Standards are size, color, and taste.

British Honduras. Juice must run out of fruit when cut in half, with the halves pressed together and held on edge. Fruit destined for canning must be tree-ripened.

Cuba. Cut fruit transversely. If juice exudes, the fruit is ripe. Ripeness is also indicated by the softness of fruit.

Cyprus. Maturity is determined by appearance of fruit.

Israel. The maturity standards for grapefruit prescribed by *Regulations Concerning the Control of the Export of Citrus Fruit* are stated as follows.

1. The minimum juice content must be 40 per cent of the total weight of the fruit.
2. The minimum ratios of T.S.S. to acid must be:

September 20 to October 10	5.1:1
October 11 to October 20	5.2:1
October 21 to October 31	5.3:1
November 1 to November 7	5.4:1
From then on	5.5:1

3. The minimum color may be greenish at the time of packing (as recommended by the European standard). The fruit may be degreened if the color is not satisfactory. (Data supplied by M. Atlas, Director of Inspection Service, Ministry of Agriculture.)

Jamaica. The fruit is regarded as mature when the ground color lightens to pale yellow-green and the rind yields appreciably to pressure.

Mozambique. Governed by the soluble-solids:acid ratio. The juice content must not be below a certain minimum.

Puerto Rico. The fruits are harvested when they are just tree-ripe; or yellow; or green, turning yellow. It all depends on the demand.

South Africa (Pretoria). Fruit is considered to be mature when hand reaming extracts a minimum of 38 per cent juice with a minimum solids:acid ratio of 5.5:1.

Southern Rhodesia. Maturity is judged by appearance only.

Surinam. Fruit is considered mature when color changes from green to yellow and when the solids:acid ratio is at least 7:1. For export, the ratio may sometimes reach 10:1.

Trinidad-Tobago. The criterion is tree-ripeness, supported by an adequate percentage of juice content.

Turkey. Solids:acid ratio of 5.5:1.

Several areas have reported, by correspondence, that they do not have any maturity standards because they do not produce grapefruit on a commercial basis. These are: Bermuda; Chile (Santiago); French Cameroun; French Equatorial Africa; Gambia, West Africa; Guadeloupe, F.W.I.; Hawaii; Indonesia; Ivory Coast, French West Africa; Nigeria; and the Philippines (Manila).

However, some of these correspondents reported that they were testing different varieties of grapefruit at their experiment stations.

Florida Citrus Code Sections Governing the Sale of Fruit and Processing of Juices

THE FLORIDA CITRUS INDUSTRY produces more citrus fruits and converts more tonnage to juice products and by-products than is the case in any other citrus-growing area. As a result of this situation, the state of Florida has created a state citrus commission, with the authority to standardize Florida's citrus industry in order to enhance the quality and reputation of its citrus fruits, including canned and concentrated products, in both domestic and foreign markets. That portion of the Florida Citrus Code (Chapter 601) which contains information concerning the standardization, for sale, of fresh grapefruit and juice products is quoted. (Florida Department of Agriculture, 1963.)

601.45 Grading of Citrus Fruit. All citrus fruit, except as provided in §601.50, intended for consumption in fresh form, sold, or offered for sale, or offered for shipment, or being shipped by common carrier or otherwise, shall be graded according to the standards established from time to time by the commission, or at the option of the shipper according to the standards as now fixed by the United States Department of Agriculture, or as such standards may be hereafter modified or changed; and all such citrus fruit shall have stamped thereon the grade thereof according to such standards, or, under such order or regulations the commission may prescribe, the shipper or seller shall have the privilege of stamping on citrus fruit either brands or trademarks which shall represent state grades or United States grades and be registered with the commission, and the commission may receive and file for record such brands or trademarks, except that, in case such citrus fruit is sold, offered for sale, or offered for shipment, or being shipped by common carrier or otherwise, enclosed in a container which meets the standards established by the commission, it shall be sufficient if the grade of said citrus fruit shall be stamped upon said closed container in the way and the manner prescribed by the commission; provided, that under such order or regulation as the commission may prescribe, the shipper or seller using closed containers shall have the privilege of using instead of marking either labels, brands, or trademarks which shall represent state grades or United States grades and be registered with the commission, and the commission may receive and file for record such labels, brands, or trademarks. For the use and benefit of those shippers desiring to have said citrus fruit graded according to the standards as now fixed by the United States Department of Agriculture, or as such standards be hereafter amended, modified, or changed, the official United States standards for citrus fruits as applied to Florida are adopted.

601.46. Conditions Precedent to Sale of Citrus Fruit. (1) It is unlawful, except as provided in §601.50, for any person to sell or offer for sale, to transport, prepare, receive, or deliver for transportation or market any citrus fruit in fresh form unless such fruit has matured in accordance with the maturity standards and is accompanied by a certificate of inspection and maturity thereof issued by a duly authorized citrus fruit inspector of the Department of Agriculture of Florida.

(2) Inspection for maturity may be made at any time, anywhere, after the fruit is severed from the tree until the shipment, after inspection and certification, is accepted by common carrier or until it has been transported beyond the state lines where being transported other than by a common carrier; and with the further proviso that shipments in bulk, either by common carrier or otherwise, to a packing house for

repacking in Florida must be reinspected and recertified before final delivery to a carrier; provided, however, that only one inspection fee shall be paid by the shipper; provided that it shall be unlawful at any time for any person to sell or offer for sale, to transport, to prepare, receive, or to deliver for transportation or market any citrus fruit which is immature or otherwise unfit for human consumption, or for any person to receive any such citrus fruit under a contract of sale, or for the purpose of sale, offering for sale, transportation, or delivery for transportation thereof; provided further, that these provisions shall not apply to sale of citrus fruit "on the trees," or to common carriers or their agents when the fruit accepted for transportation or transported by any common carrier is accompanied by a proper certificate of maturity and grade of such fruit.

601.47. Condition Precedent to Canning or Concentrating Citrus. It is unlawful for any person to can any citrus fruits or to can or concentrate the juices thereof unless such fruit is mature in accordance with the maturity standards and is accompanied by a certificate of inspection and maturity thereof issued by a duly authorized citrus fruit inspector of the Department of Agriculture of Florida. Inspection for maturity shall be made at the canning or concentrating plant, with the further proviso that shipments either by common carrier or otherwise to a canning plant or a concentrating plant in Florida must be reinspected and recertified before use by the canner or concentrator.

601.48. Grading of Canned or Concentrated Citrus Fruit Products. All canned or concentrated products of citrus fruit, except as provided in §601.50, sold, or offered for sale, or offered for shipment shall be graded according to the standards established from time to time by the commission, or at the option of the shipper according to the standards as now fixed by the United States Department of Agriculture, or as such standards may be hereafter modified or changed, and the immediate containers of such canned or concentrated products shall have the grade stated thereon in such manner as the commission may prescribe by rule or regulation, but provided, however, that if such canned or concentrated products shall meet the requirements of the two highest grades according to the standards established from time to time by the commission, or at the option of the shipper the two highest grades according to the standards as fixed by the United States Department of Agriculture, or as such standards may be hereafter modified or changed, the shipper shall have the privilege of using either labels, brands, or trademarks which shall represent such state grades or United States grades and be registered with the commission in lieu of stating the grade on the immediate container, and the commission shall receive and file for record such labels, brands, or trademarks.

601.49. Condition Precedent to Selling Canned or Concentrated Citrus Fruit Products. It is unlawful for any person, except as provided in §601.50, to sell or offer for sale, to transport, receive, or deliver for transportation or market any canned or concentrated product of citrus fruits unless the same has been inspected and is accompanied by a certificate of inspection issued by a duly authorized inspector of the Department of Agriculture of Florida, provided, however, that the commission shall by regulation provide that in lieu of the accompaniment of such shipment by a certificate of inspection, the fact of such inspection may be shown by appropriate means on the manifest or bill of lading covering such shipment.

601.50. Sale or Shipment of Citrus or Citrus Products for Certain Purposes Exempt. Irrespective of the provisions of §§601.45, 601.46, 601.48, 601.49, 601.51, and 601.52, the commission, under such precautionary rules and regulations as it may deem expedient, shall permit sale or shipment of citrus fruit or the canned or concentrated

products thereof without the issuance of and filing of inspection certificate and without the grade being shown on the container thereof, of:

(1) Intrastate shipments of fresh citrus fruit for consumption or use within the state.

(2) Shipments to be used for charitable or unemployment relief purposes.

(3) Shipments to the United States government or any of its agencies and interstate shipments to any packing house, canning plant, or concentrate plant for commercial processing, as may be defined by the commission; or to fresh fruit juice distributors outside the state;

(4) Shipments by any method of transportation by "gift fruit shippers," as defined by the commission, but such shipments shall not be for the purpose of resale by the consignee thereof.

But provided, however, that no such rule or regulation issued hereunder shall permit or allow the sale or shipment of citrus fruit deemed by this act to be immature and unfit for human consumption or of canned or concentrated products thereof prepared or made from citrus fruit deemed by this law to be immature and unfit for human consumption; but provided further that shipments under subsections (1) and (4) above shall meet such minimum grade standards as may, from time to time, be established by the Florida citrus commission; and provided further that such rules and regulations shall provide for the due collection of any advertising taxes and inspection fees that may be due thereon.

601.51. Shipments of Citrus Fruit or Products. No common carrier, or other carrier, or person, except as provided in §601.50, shall accept for shipment, ship, or transport any citrus fruit or the canned or concentrated products thereof until a grade certificate is issued showing the grade thereof, which certificate or a duplicate thereof shall be filed with the carrier at the point of shipment, nor shall any common carrier, or other carrier, or person accept for shipment or ship any citrus fruit or the canned or concentrated products thereof where written notice has been given to such common carrier, or other carrier, or person, or his representative or agent by the commissioner or his authorized agent, employee, or inspector that said citrus fruit or the canned or concentrated products thereof does not comply with the provisions of the law or the rules and regulations promulgated by the commission or the commissioner; provided that the shipper or handler of such citrus fruit or the canned or concentrated products thereof shall have the privilege of repacking or remarking, and that if or when the same shall have been repacked or remarked to conform to the provisions of law or said rules, regulations, or orders promulgated by the commission or the commissioner, the commissioner or his authorized inspector or agent shall notify said common carrier or other carrier, or person, or his agent that such citrus fruit or the canned or concentrated products thereof may be accepted for shipment, and such shipper or handler shall not be considered as having violated this chapter or said rules, regulations, or orders, but provided further that this section shall be deemed to have been complied with if the shipper shall have conformed to regulations issued by the citrus commission under the provisions of §601.49.

601.52. Carriers Are Not to Accept Fruit Unless Same Bears Evidence of Payment of Excise Taxes. No common carrier or other carrier or person, except as provided in §601.50, shall accept for shipment, or ship, or transport any citrus fruit, or the canned or concentrated products thereof, unless the grade certificate, manifest, or bill of lading covering said citrus fruit or the canned or the concentrated products thereof bears evidence of the payment, as provided by law, of the advertising excise tax provided for in §601.15, and of the payment of inspection tax, as provided by §601.28, and, if applicable, the excise tax for color-add, as provided by §601.83.

601.53. Unlawful to Can or Concentrate Unwholesome Citrus. It is unlawful for any person to can or concentrate, or buy for canning or concentrating purposes, or sell for canning or concentrating purposes in Florida any citrus fruit that is unwholesome or decomposed so that it is unfit for canning or concentrating purposes.

601.54. Seizure of Unwholesome Fruit. (1) The commissioner or his duly authorized inspectors shall seize and destroy all citrus fruit found by said commissioner or inspectors to be unwholesome or decomposed so that it is unfit for canning or concentrating purposes as defined by law or by any regulation of the commission pursuant to authority given in this chapter and, in the event any inspector shall find that any canner or concentrator is canning or concentrating fruit prohibited to be used, he may seize and destroy not only such fresh fruit found in the canning or concentrating plant but also citrus fruit or juice in the process of being canned or concentrated or which has been canned or concentrated from the same lot or shipment wherein the fresh fruit is found by said inspector to be subject to seizure under the provisions of this section.

(2) Whenever any inspector finds citrus fruit in the canning or concentrating plant which should be destroyed under the provisions of this law, the operator, manager, or other person in charge of the canning or concentrating plant shall make known to the inspector the code number or other manner of identifying any fruit or the canned or concentrated products thereof that has been canned or concentrated from the same lot or shipment wherein is found the said fruit subject to be seized.

601.0105. Canned Grapefruit Juice; Standards; Labeling. No canned grapefruit juice shall be sold or offered for sale or shipped or offered for shipment which:

(1) Is prepared from raw juice containing, before the addition of any additive, less than seven and five-tenths per cent total soluble solids; or

(2) When canned, contains less than nine per cent total soluble solids; or

(3) Has a ratio of total soluble solids to anhydrous citric acid of less than seven and one-half to one; or

(4) Contains less than 0.75 per cent anhydrous citric acid; or

(5) Contains more than 0.020 per cent recoverable oil; or

(6) Does not meet requirements to be established by the commission regarding color, absence of defects, taste, and flavor; unless the immediate container thereof shall be labeled in accordance with regulations of the commission and there shall appear on such label the word "substandard" in bold type not less than one-fourth inch high printed or stamped diagonally thereon.

601.0106. Canned Blended Juice; Standards; Labeling. No canned blend of orange and grapefruit juice shall be sold or offered for sale or shipped or offered for shipment which:

(1) Is prepared from mixed raw juice of oranges and grapefruit containing before the addition of any additive less than eight per cent total soluble solids; or

(2) When canned, contains less than nine and five-tenths per cent total soluble solids; or

(3) Has a ratio of total soluble solids to anhydrous citric acid of less than eight to one; or

(4) Contains less than 0.65 per cent or more than 1.80 per cent anhydrous citric acid; or

(5) Contains more than 0.040 per cent recoverable oil; or

(6) Contains when mixed and before canning more or less than the percentage of orange juice determined by rule or regulation of the commission required to be contained therein, and does not meet requirements to be established by the com-

mission regarding color, absence of defects, taste, and flavor; unless the immediate container thereof shall be labeled in accordance with regulations of the commission, and there shall appear on such label the word "substandard" in bold type not less than one-fourth inch high printed or stamped diagonally thereon.

601.0108. Frozen Concentrated Orange Juice; Requirements; Labeling. No frozen concentrated fresh orange juice shall be sold, or offered for sale, or shipped, or offered for shipment which:

(1) Is concentrated to less than 41.8 or more than 44 degrees Brix. The Brix reading, if determined refractometrically, shall include corrections for citric acid; or

(2) Has a lower ratio of total soluble solids to anhydrous citric acid of less than twelve to one or a higher ratio of total soluble solids to anhydrous citric acid than nineteen to one; or

(3) Contains more than 0.120 milliliter of recoverable oil per 100 grams of concentrate; or

(4) Contains any additives of any kind; or

(5) Does not taste essentially the same as freshly expressed orange juice of similar quality and is not completely free of all fermented, cooked terpeney, or other off-flavors; or does not meet requirements to be established by the commission regarding color, absence of defects, taste, and flavor; unless the immediate container thereof shall be labeled in accordance with regulations of the commission, and there shall appear on such label the word "substandard" in bold type not less than one-fourth inch high printed or stamped diagonally thereon.

(6) Provided, however, that this section shall not prohibit the sale or shipment of 4 plus 1 (five-fold) frozen concentrated orange juice which meets the foregoing requirements, except the Brix requirements of subsection (1), in institutional size containers. The commission may, by rule or regulation, define institutional size containers and establish the Brix range for 4 plus 1 (five-fold) frozen concentrated orange juice.

Although the chemical composition and the physical and chemical properties of grapefruit of good quality in either the fresh or processed form are discussed in this book in relation to other factors, it is important to note that these same fruit characteristics are the underlying factors on which the regulatory measures quoted in the foregoing text are based. Consequently, external color, flavor, and odor, soluble solids, titratable acidity and pH, Brix:acid ratio and ascorbic acid are characteristics of grapefruit that are measures of quality, not only for fruit destined for the fresh market, but they are also applicable to fruit diverted to juice processing. Other factors, however, that are specifically important in the quality of canned and frozen grapefruit juices are pulp content, cloud stability, gelation, oxygen content, oil content, pectinesterase activity, glycoside content (naringin) and, in some instances, consistency or viscosity. These intrinsic characteristics of grapefruit are certainly the bases of arriving at high-quality fruit that can be consumed either fresh or processed.

Chapter 5

Postharvest Changes in Grapefruit

GRAPEFRUIT AND ORANGES are not held in storage to the same extent as are deciduous fruits, but are "stored" on the trees until they are ripe, as pointed out by Miller (1946*b*). However, he noted that there would be a few exceptions to this statement. For example, when the citrus fruits are picked, the market may not be ready for all of the crop, and the excess must be stored in packing houses before shipment, or else stored at the market end of the line, if the quantity is too great for immediate consumption. Grapefruit cannot be stored on the trees too long—not more than several weeks or possibly a very few months—or they may begin to regreen or show physiological or pathological breakdown.

Temperature and Humidity in Storage and Transit

THE CHOICE of an optimum storage temperature depends on the type of citrus fruit, and involves a compromise between preventing decay, which occurs at a relatively high temperature, and preventing physiological disorders, which occur at relatively low temperatures.

The disorders commonly found in stored fruit are usually referred to as low-temperature injuries. However, there are certain factors that exert an influence on the susceptibility of the fruit to low-temperature breakdown. For example, fruits produced under irrigation in areas of insufficient rainfall (California, Arizona, Texas, Spain, Italy, Israel, Australia, and all except the coastal areas of South Africa) are not so susceptible to stem-end decays as are those grown in regions having ample rainfall (Florida, South America, Japan, China, and New Zealand). In addition to irrigation and abundant rainfall, soil type, climate, cultural practices, position of the fruit on the tree, maturity of fruit when picked, and temperature for several days preceding harvest are known to influence the storage life. The effects of these factors may help to explain the great diversities in results that have been reported by workers in different regions, not only in the United States but in other parts of the world where grapefruit is grown.

The temperature at which grapefruit is stored may affect not only its external appearance, but also its composition. Zoller (1918*a*), Hawkins and Magness (1920), and Hawkins (1921) were among the first to study this.

Stahl and Camp (1936) were among the early workers who studied the effects on Silver Cluster and Marsh grapefruit of storage temperatures ranging from 32 to 80° F. (zero to 27° C.). The results on the Marsh variety are shown in tables 168 and 169. The results for Silver Cluster were similar to those for Marsh, except that Silver Cluster showed a little less decay and had a slightly better general appearance, and the specific gravity increased slightly. Considering all points—loss in weight, decay, pitting, keeping quality before and after removal from storage, taste, marketability at any one time—the best temperatures for storage of unwrapped, untreated Silver Cluster and Marsh grapefruit were from 37.5 to 42° F. (3 to 5.5° C.), preferably 37.5°.

Harvey and Rygg (1936*c*) determined some of the chemical changes that occurred in the peel of Marsh grapefruit in storage at different temperatures. The experiments were performed on fruit grown in the Corona-Fontana area of California (intermediate between coast and desert) and Oasis (in the desert area). For each storage temperature, the data reported for the Corona-Fontana samples were means for the over-all area and for all portions of the peel (table 170). For the Oasis samples, the averages were for all collections and all portions of the peel.

Owing to the high relative humidity of the storage rooms, the moisture content of the peel samples changed only slightly during storage. In all instances, the total sugars decreased, with the least drop at 32° and the greatest at 52° F. (zero and 11° C.). The amount of invert sugar in the peel decreased at all storage temperatures. The rate of inversion was approximately the same in the peel from both districts, with slight activity at 32° and a more rapid inversion at 42 and 52° F. (5.5 and 11° C.). In the Corona-Fontana fruit, the reducing sugars of the peel increased at 42 and 52°, with an insignificant decrease at 32°. In the Oasis fruit, they increased at all storage temperatures. The slight increase at 32° was attributed to the polysaccharides; the invert sugar produced from sucrose at that temperature was probably utilized for

TABLE 168

SPECIFIC GRAVITY OF MARSH GRAPEFRUIT STORED AT VARIOUS TEMPERATURES FOR 4 MONTHS.*

Dates (1933)	Specific gravity after storage at degrees F. and C.						
	70–80° (21–27°)	58° (14.4°)	54° (12°)	48° (9°)	42° (5.5°)	37.5° (3°)	32° (zero)
April 6†.....	0.8672	0.8672	0.8672	0.8672	0.8672	0.8672	0.8672
June 1 or 2.....	.8659	.8627	.8849	.8669	.8460	.8420	.8652
July 7 or 8.....	(Decayed)	.8654	.8620	.8628	.8609	.8540	.8538
August 4 or 5..	.....	(Decayed)	.8626	.8560	.8570	.8366	.8590

*Adapted from Stahl and Camp (1936).
†Picking date; lots in this line were nonstored controls.

TABLE 169

CHANGES IN COMPOSITION OF MARSH GRAPEFRUIT STORED AT VARIOUS TEMPERATURES FOR 4 MONTHS.*

Storage temperatures and dates of analyses (1933)	Specific gravity of fruit	Weight of fruit (grams)	Per cent juice	Specific gravity of juice	Per cent acid (citric)	pH	Per cent sugars (as dextrose)		
							Free reducing	Hydro-lyzable	Totals
70–80° F. (21–27° C.)									
April 6	0.8672	554.4	56.6	1.040	2.0	3.0	3.29	1.90	5.19 (check)
June 1	.8659	481.4	55.9	1.041	1.9	3.2	3.57	1.98	5.55
July 9†	.....	.....	....	.....	...	...	....	....	....
58° F. (14.4° C.)									
April 6	.8672	554.4	56.6	1.040	2.0	3.0	3.29	1.90	5.19 (check)
June 2	.8627	500.8	53.4	1.042	1.9	3.8	3.57	2.37	5.94
July 9	.8654	475.5	54.5	1.043	1.9	3.5	3.46	2.72	6.18
August 4†	.....	.....	....	.....	...	...	....	....	....
54° F. (12° C.)									
April 6	.8672	554.4	56.6	1.040	2.0	3.0	3.29	1.90	5.19 (check)
June 2	.8849	412.1	55.7	1.045	2.2	3.9	3.58	2.30	5.88
July 8	.8620	422.2	56.2	1.049	2.1	3.9	3.50	2.00	5.50
August 4	.8626	350.3	52.3	1.051	2.0	3.5	3.63	3.12	6.75
48° F. (9° C.)									
April 6	.8672	554.4	56.6	1.040	2.0	3.0	3.29	1.90	5.19 (check)
June 1	.8669	430.8	54.9	1.042	1.9	3.8	3.40	1.90	5.30
July 9	.8628	419.5	54.4	1.045	2.1	3.4	3.35	2.32	5.67
August 5	.8560	436.2	55.1	1.044	1.9	3.2	3.35	3.20	6.55
42° F. (5.5° C.)									
April 6	.8672	554.4	56.6	1.040	2.0	3.0	3.29	1.90	5.19 (check)
June 2	.8460	432.1	53.8	1.043	2.1	4.0	3.32	2.55	5.87
July 8	.8609	465.0	55.8	1.040	1.7	3.4	3.30	2.88	6.18
August 5	.8570	425.1	54.3	1.044	1.9	3.1	3.40	2.55	5.95
37.5° F. (3° C.)									
April 6	.8672	554.4	56.6	1.040	2.0	3.0	3.29	1.90	5.19 (check)
May 30	.8420	428.2	56.1	1.043	1.9	3.3	3.20	2.25	5.45
July 8	.8540	398.8	53.5	1.045	2.1	3.3	3.98	2.80	6.78
August 4	.8366	436.0	51.5	1.047	2.0	2.9	3.42	2.90	6.32
32° F. (zero C.)									
April 6	.8672	554.4	56.6	1.040	2.0	3.0	3.29	1.90	5.19 (check)
May 30	.8652	438.7	57.1	1.045	1.9	3.5	3.58	3.14	6.72
July 8	.8538	475.8	50.6	1.040	1.9	3.8	3.89	3.00	5.89
August 4	.8590	413.3	52.8	1.043	1.8	3.4	2.90	2.95	5.85

*Adapted from Stahl and Camp (1936).
†All fruit was decayed.

TABLE 170

EFFECTS OF STORAGE TEMPERATURES ON THE COMPOSITION OF PEEL OF MARSH GRAPEFRUIT GROWN IN DIFFERENT LOCALITIES IN CALIFORNIA.*

Localities and storage temperatures (F. and C.)	Water (per cent of fresh weight)	Constituents (percentage of dry weight)							Ratio of flavedo to albedo	pH		Acidity (ml of *N* acid per 10 ml of pulp juice)
		Reducing sugars	Invert sugars	Total sugars	Naringin	Hydrolyzable polysaccharides	Soluble solids	Insoluble solids		Of peel	Of pulp juice	
Corona-Fontana:†												
No storage	80.1	24.58	12.91	37.49	8.03	7.28	70.5	29.5	0.76	6.40	3.09	3.33
52 (11)	79.1	29.52	2.69	32.21	7.40	7.56	68.1	31.9	0.76	6.10	3.08	3.30
42 (5.5)	79.6	30.33	3.60	33.93	6.91	7.41	68.4	31.6	0.71	5.97	3.06	3.30
32 (zero)	79.5	23.85	11.40	35.25	6.38	7.44	69.6	30.4	0.72	6.29	3.09	3.17
Oasis:‡												
No storage	76.8	20.67	9.69	30.36	6.47	11.62	61.0	39.0	...	6.23	3.28	2.48
52 (11)	77.0	22.89	2.69	25.58	7.73	11.63	58.9	41.1	...	5.90	3.35	2.56
42 (5.5)	76.1	23.45	3.65	27.10	7.95	11.33	59.8	40.2	...	5.91	3.28	2.50
32 (zero)	76.0	21.86	8.06	29.92	8.17	11.13	61.7	38.2	...	6.04	3.33	2.43

*Adapted from Harvey and Rygg (1936c).
†Each figure is the average of 32 determinations.
‡Each figure is the average of 40 determinations.

respiration during the storage period. In the Corona-Fontana fruit, the naringin of the peel decreased at all temperatures; in the Oasis fruit, it increased at all storage temperatures. In all instances, the pH of the peel of stored fruit was lower than that of the control samples.

Martin *et al.* (1939) picked Arizona grapefruit samples at monthly intervals from December to June, and studied the effects of storage on the acid and soluble-solids contents of the juice. The fruit samples were washed and waxed in the usual commercial manner, and placed in air-conditioned storage rooms at a temperature of 60° F. (16° C.) and relative humidity of 88 per cent. Figure 126 shows the changes in percentage of acid in stored grapefruit as influenced by the date of picking from the grove and by the length of the storage period. The percentage of citric acid in the juice increased slightly in the first months of storage; but with prolonged storage, continual decreases were noted, and the total acidity reached values far below the original concentration. The heavy continuous line in the graph shows the decrease in acid content of fruit left on the tree; in late samplings, a low acid percentage was noted.

MacRill *et al.* (1946) noted that grapefruit with less than 1 per cent acid (as citric) would not be considered of good storage quality, and that when the titratable acidity reached 0.5 per cent, the fruit should be shipped to market immediately, or disposed of through other channels.

Martin *et al.* (1939) further determined that the total soluble solids of the juice of grapefruit generally increased for a time in storage, but eventually

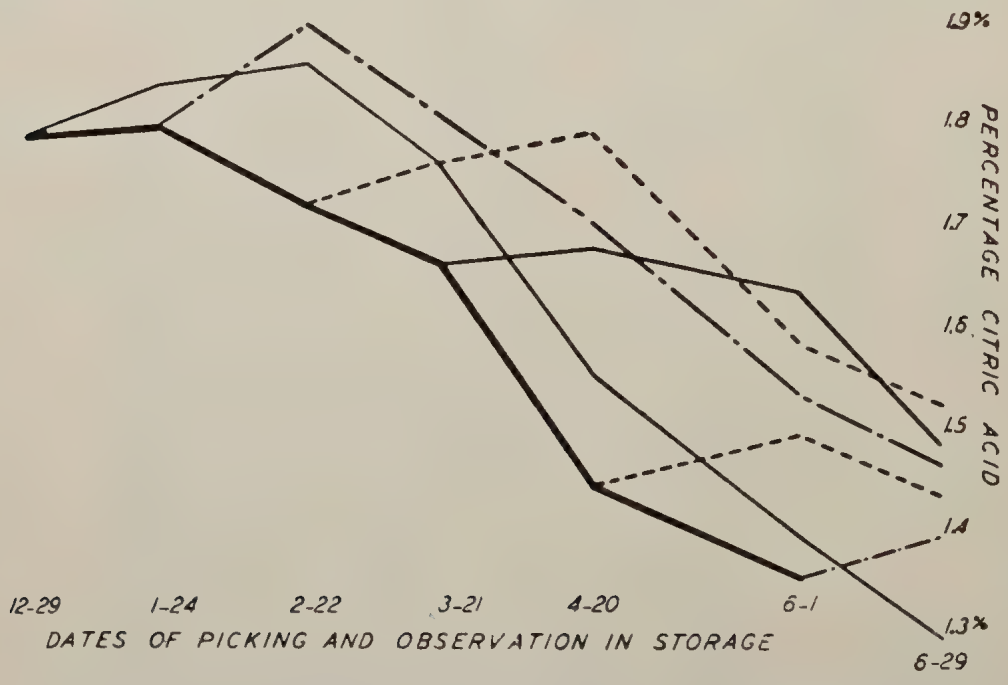


Fig. 126. Effects of picking dates on the percentage of citric acid in stored grapefruit. The heavy line indicates the decrease in acid content of juice of fruit remaining on the trees. (From Martin *et al.*, 1939.)

decreased after longer storage. In none of the samples did the total soluble constituents reach so low a point as in fruit that remained on the tree after the maximum concentration of total soluble solids had been reached (figure 127). Note that the total soluble solids of field samples (heavy continuous line) reached a maximum on February 22 for the 1939 season, while a continuous decrease occurred in the fruit picked after that date. With respect to the changes in the concentrations of acids and total soluble solids, the data seem to indicate that the reactions that take place in grapefruit left on the tree may occur also in stored fruit, but at a much slower rate.

No general statement can be made as to what is the best storage temperature for grapefruit in the various citrus-growing regions. With respect to atmospheric moisture in the storage room, the data seem to indicate that 85 to 90 per cent relative humidity is most desirable. The lack of agreement on a uniform temperature for grapefruit storage is undoubtedly caused by many important factors, such as: variety; climatic and environmental conditions under which the fruit is grown; cultural and pest-control programs of the grove; picking under adverse weather conditions; degree of maturity at time of picking; and packing-house operations, such as cleaning, treating, waxing, grading, and final packing. It should be noted that fruits grown in Arizona and California have a tendency to become highly colored or bronzed when stored below 60° F. (16° C.), and that they deteriorate rapidly when stored above that temperature.

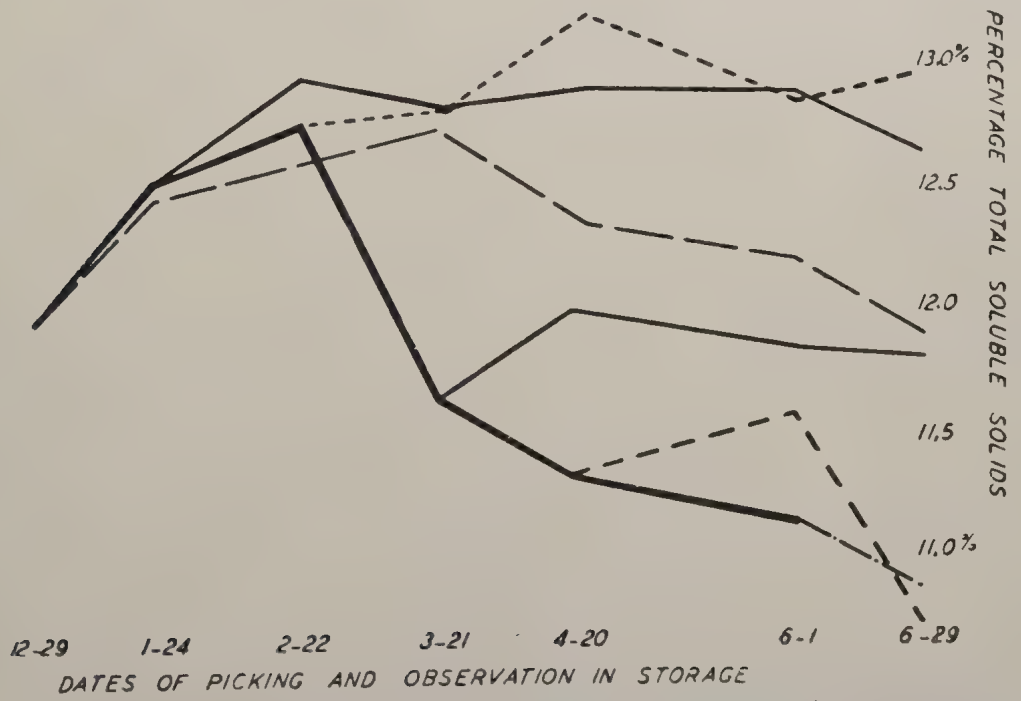


Fig. 127. Effects of picking dates on the percentage of total soluble solids in stored grapefruit. The heavy line indicates the changes of total soluble solids of field samples during the season. (From Martin *et al.*, 1939.)

It is well known that the condition in which the fruit arrives at its destination depends greatly upon its temperature after harvest, both before and during transit. As a result of metabolic activity, the tissues of grapefruit gradually become overripe and break down. These changes may be retarded, and decay checked, by reducing the temperature of the fruit promptly after harvest, and maintaining it at a relatively low temperature until used.

Van der Plank *et al.* (1938) reported that the maximum breakdown of grapefruit in cold storage usually occurred at intermediate temperatures of 40 to 45° F. (4.4 to 7.2° C.), but that certain lots of fruit might suffer greatest injury at other temperatures, such as 30 to 50° F. (–1.1 to 10° C.) .

Van der Plank and Rattray (1940) found that grapefruit of good keeping quality suffered very little decay, even when stored at 60° F. (16° C.) in boxes lined with waxed crystalline paper. Slight and inconsistent changes in the percentages of soluble solids in the juice were found during storage at 40° F. (4.4° C.). (See also, Van der Plank and Rattray, 1938*a, b.*)

E. G. Hall (1938), in Australia, stated that grapefruit was particularly susceptible to the disorder known as "storage spot," and kept best at temperatures higher than those that were most suitable for storing oranges.

Martin *et al.* (1939), in Arizona, noted that waxed and carefully handled grapefruit could be stored for at least 3 months at 60° F. and 88 per cent relative humidity if it was harvested before the spring flush of growth in April; but if picked after that time, its storage life became increasingly shorter, probably because of decreases in total solids and citric acid.

Wardlaw *et al.* (1934) observed the relative effects of 45 and 50° F. (7.2 and 10° C.) on the start of fungal growth and decrease in moisture content.

Leonard (1936) and Leonard and Wardlaw (1937) , in studying the effects of temperature on stored grapefruit, reported that prolonged cold storage caused a marked loss of flavor, but only a slight change in sugar content and in titratable acidity.

Wardlaw and Leonard (1939) stated that in the British West Indies, the chilling temperature for "wet-season" fruit was very close to 45° F. (7.2° C.), the temperature then being used for storage. It was found inadvisable to use air at 40° F. (4.4° C.) for rapid cooling for more than the first 12 to 15 hours.

Van der Plank *et al.* (1938), in South Africa, noted that to ensure against cold injury, the storage temperatures for grapefruit should be as high as 50 to 55° F. (10 to 13° C.). These temperatures, especially early in the season, were also beneficial with respect to flavor and texture. As the season advanced, successively lower storage temperatures were found to be advisable.

Huelin (1942), in his report on factors affecting the storage of grapefruit and other citrus fruits in Australia, stated that in testing fruit from each of 4 districts from 1935 to 1937, he found the storage life to be much longer at 50° F. than it was at 40 or 45°.

Tindale (1944, 1945) reported that all peel disorders of Australian grapefruit were prevented by storage at 55° F. (13° C.). After 3 months' storage, the fruit was very juicy and the flavor was excellent.

Ryall and Buford (1947) found that temperatures of 46 to 50° F. were better for the storage of Texas grapefruit than 32 to 34°, or 38 to 40° (7.8 to 10; zero to 1; 3.3 to 4.4° C.). The maximum storage period ranged from 1 to 2 months. The fruit of the first picking remained in good condition longer than that of the second picking.

Hendrickson and MacRill (1948) stated that the most desirable storage temperatures for California and Arizona grapefruit were 58 to 60° F. (14.4 to 16° C.), and that the best relative humidities were 86 to 88 per cent. However, they noted that in areas where stem-end rot was prevalent, the temperatures should range from 32 to 34° F. (zero to 1° C.), even though the storage period would have to be short.

Rose *et al.* (1949) reported that for best results, grapefruit in storage should be held at 32° F. in relative humidities of 85 to 90 per cent. Under these conditions, they found that the fruit could be stored for a maximum of 10 weeks, provided that it was sound but not overripe when stored. Leggett and Sutton (1951), in Florida, gave the same figures for sound, mature fruit, but found that overmature fruit was likely to pit and decay within 8 weeks under such conditions.

W. T. Pentzer[1] has supplied the author with the data graphically illustrated in figure 128, showing the temperatures of grapefruit in transit from Cocoa, Florida, to Seattle, Washington. As a generalization, the rate of cooling in transit varies directly, not only with the initial fruit temperature, but with the difference in temperature between the fruit and that of the outside air. The lower the fruit temperature at the time of loading, the smaller the amount of ice that will be utilized in transit, given the same outside air temperature. The typical temperature responses of grapefruit during the transit period are shown in the figure; the outside air temperatures fluctuate from 86 to 10° F. (30 to –12° C.). After the third day, the outside air temperature is shown to be mostly 50° F. (10° C.) or lower. Relatively low temperatures at loading and low outside temperatures during most of the period en route should result in a proportionate decrease in ice meltage.

As noted earlier in this chapter, the most important factor in protecting fruit quality after harvest is the selection of proper storage temperatures. In studying the factors affecting the quality of grapefruit exported to overseas markets, Chace *et al.* (1966) correlated the susceptibility of grapefruit to rind breakdown and fruit decay with storage temperature, and secondly with the degree of fruit maturity. They noted that treatments such as degreening and application of fungicides and waxes, as well as handling procedures before

[1] W. T. Pentzer, United States Department of Agriculture. Unpublished data.

shipment, would affect the condition of the fruit on arrival at terminal markets and during the marketing period. For 3 seasons (1961–62 through 1963–64), these authors carried out experiments on Marsh Seedless and Ruby Red grapefruit stored under simulated export and market conditions. Fruit samples of these varieties were harvested in October and December and in March and May during 1961–62, and in October, February, and April during the next 2 crop seasons (1962–63 and 1963–64) from the same 2 groves in Indian River and Lake counties, Florida. The samples were harvested at 3 different periods: early-season fruit (October), midseason (mid-February to early March), and late-season (April or May).

The histograms of figures 129 and 130 show the effects of transit temperatures and degrees of maturity of Marsh Seedless and Ruby Red grapefruit on rind breakdown and decay of mid- and late-season samples. The juice from midseason fruit had a mean soluble-solids:acid ratio of 9.5:1. As shown in figure 129, only small variations occurred in the amount of rind breakdown that developed in midseason fruit held at 32, 50, and 60° F. (zero, 10, and 16°

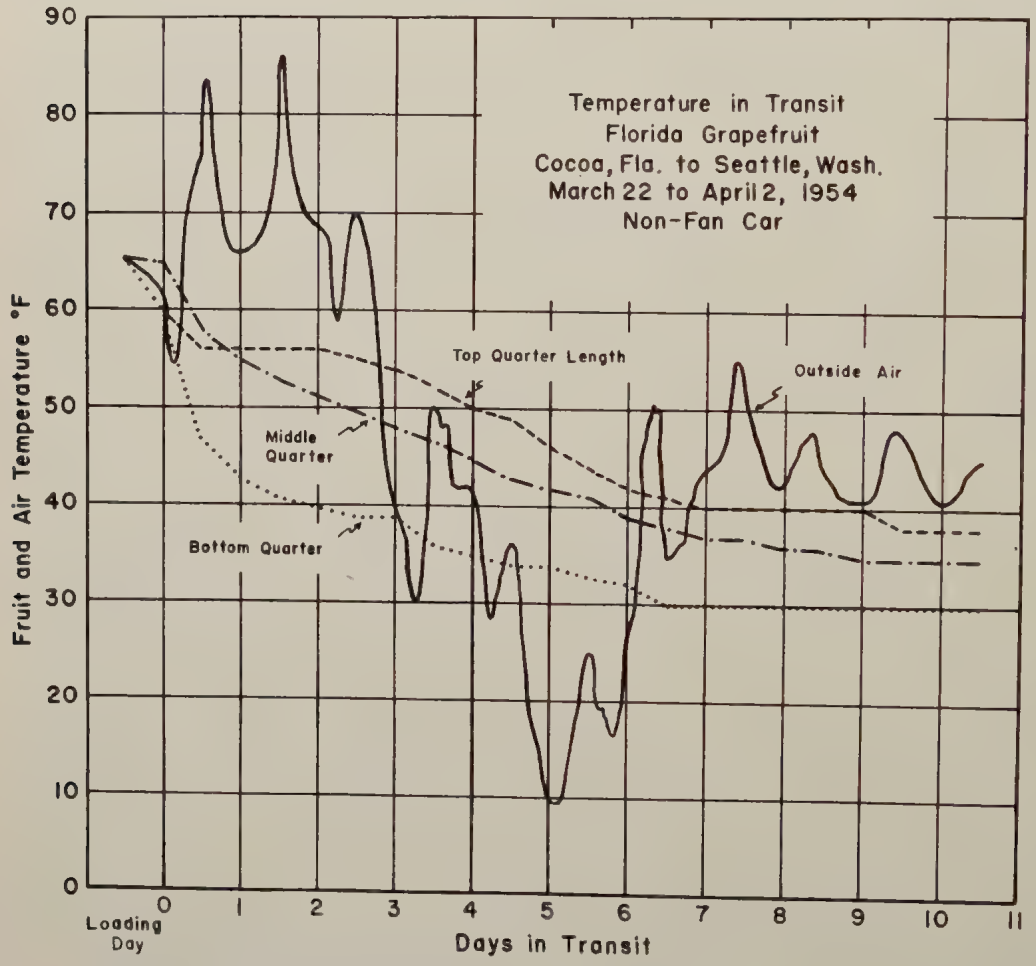
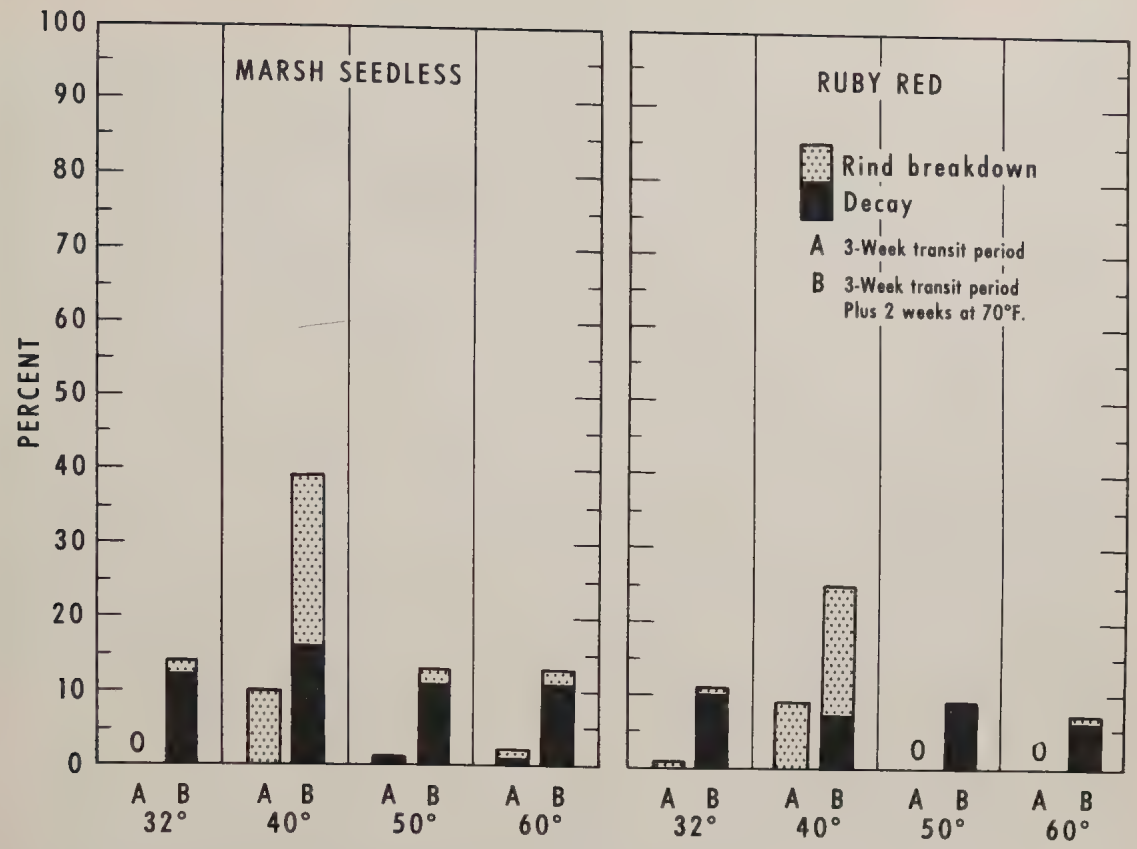
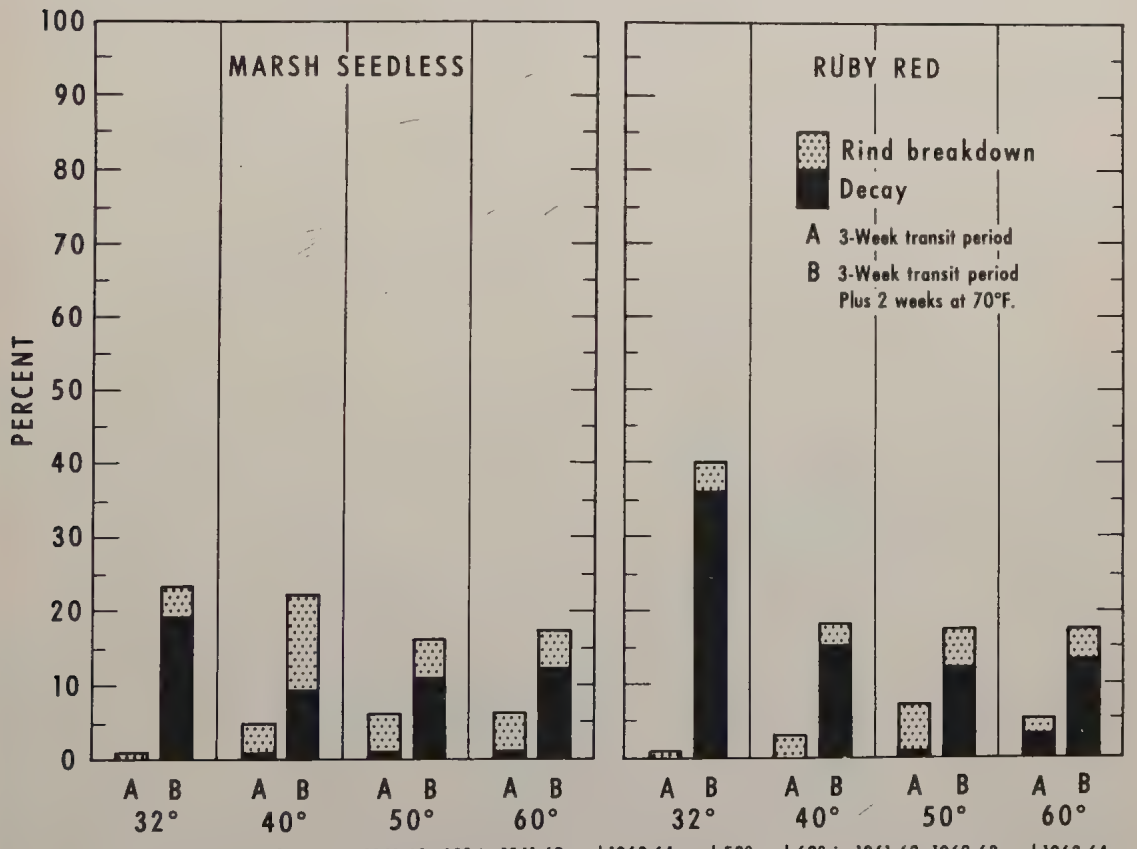


Fig. 128. Temperature responses of Florida grapefruit during a transit period.



Data are means of tests for 32° in 1961-62; 40° in 1961-62 and 1963-64; and 50° and 60° in 1961-62, 1962-63 and 1963-64.



Data are means of tests for 32° in 1961-62; 40° in 1961-62 and 1963-64; and 50° and 60° in 1961-62, 1962-63 and 1963-64.

Figs. 129 (top) and 130 (bottom). Rind breakdown and decay during simulated export tests. *Top:* Midseason fruit. *Bottom:* Late harvest. (From Chace *et al.*, 1966.)

C.). Rind breakdown in midseason samples included both pitting and aging. Significant amounts of rind breakdown occurred in fruit held at 40° F. (4.4° C.), and the amounts increased noticeably during the 2-week holding period at 70° F. (21° C.). Midseason grapefruit had a higher percentage of decay than early-season fruit at most temperatures, but the greater part of this decay developed during the 2-week holding period at 70°. Fruit that had been held earlier at temperatures of 50 and 60° F. developed 6 to 11 per cent decay dur-

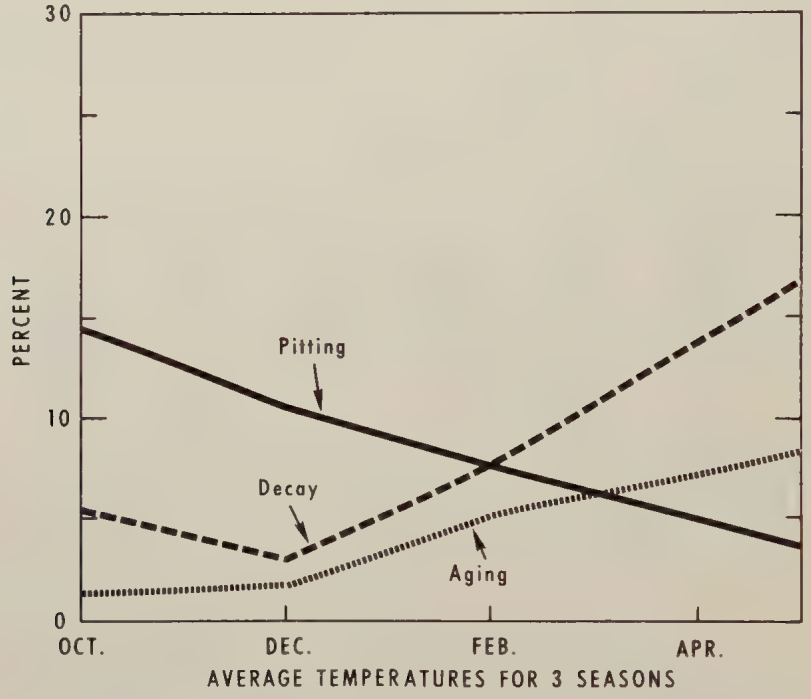


Fig. 131. Susceptibility of grapefruit, harvested at 4 maturities, to pitting, decay, and aging after a 3-week storage period at various temperatures and 2 weeks at 70° F. (21° C.). (From Chace *et al.*, 1966.)

ing the holding period, while samples held earlier at 32 and 40° F. showed similar variations in decay from 7 to 16 per cent. Marsh Seedless showed more decay than Ruby Red.

Figure 130 shows the effect of temperature on rind breakdown and fruit decay of late-harvest grapefruit under simulated export conditions. The fruit samples (including both varieties) had an average total soluble-solids:acid ratio of 12:1. The rind breakdown present on the late-harvest fruit was classed as aging. Storage temperatures during the simulated 3-week transit period were not effective in controlling this type of rind breakdown. In the late-season fruit, the degree of aging that developed during the transit period was associated with greater amounts of fruit decay during the 2-week holding period at 70° F. It is noteworthy that only slight decay occurred during the

3-week transit period (see histograms at *A*), but highly significant amounts developed during the 2 weeks at 70° F. (see histograms at *B*). Grapefruit stored at 32° F. (zero C.) had 19 and 36 per cent decay, which was higher than the amounts reported for other storage temperatures.

It has been fairly well established that many physical and chemical characteristics occurring in fruits during growth and maturation are reflected to a considerable degree in the responses of postharvest fruits when they are subjected to various storage and transit temperatures and humidities. This is borne out by the data illustrated in figure 131, which show the relationships of pitting, aging, and fruit decay to the degree of maturity of grapefruit. For the 3-year experimental period, the percentages of these 3 types of rind breakdown were plotted against the dates when the samples were harvested, without reference to their transit or storage temperatures. The early-season fruit developed the highest percentage of pitting, and the susceptibility to pitting gradually decreased with increased fruit maturity. The time-course curves for aging and decay are in direct contrast to the one for pitting, indi-

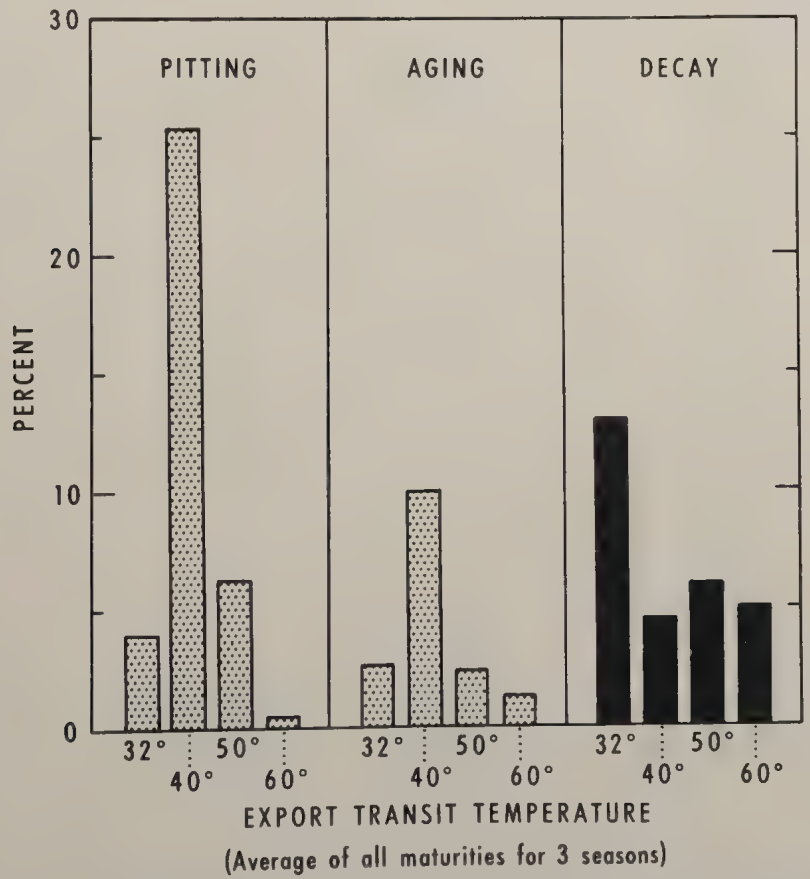


Fig. 132. Susceptibility of grapefruit to pitting, aging, and decay after a 3-week storage period at various temperatures and 2 weeks at 70° F. (21° C.). (From Chace *et al.*, 1966.)

cating that aging and decay increased with fruit maturity. With the increase in aging characteristics of the fruit tissues with maturity, a corresponding increase in decay occurred as a result of the aged fruit being more susceptible to both physiological and pathological contributing factors.

Figure 132 shows the effects of storage temperatures on rind breakdown without reference to fruit maturity. Consequently, average values for pitting, aging, and decay for all maturities for 3 seasons were obtained for each transit temperature. Note that the amount of pitting was definitely associated with the storage temperature. The highest percentage of pitting occurred in grapefruit stored at 40° F., and the lowest occurred at 60°. Fruit stored at 40° had the greatest susceptibility to aging, but this did not develop to any serious extent at 32, 50, and 60°. The highest percentage of decay developed in

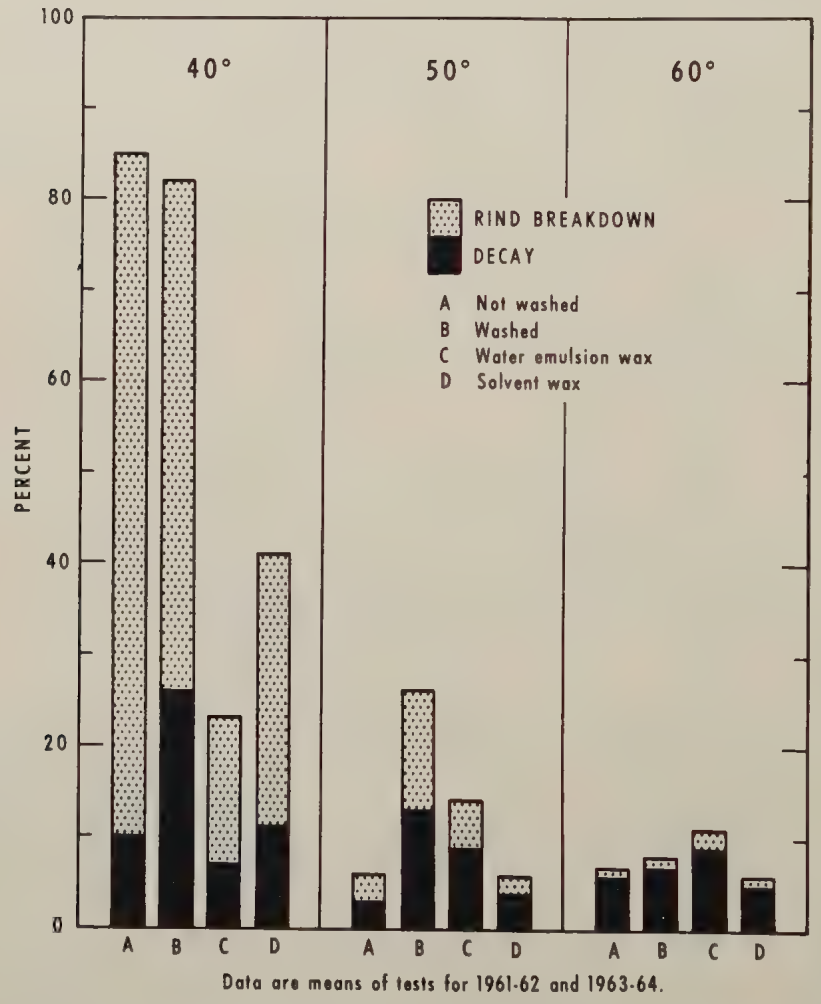
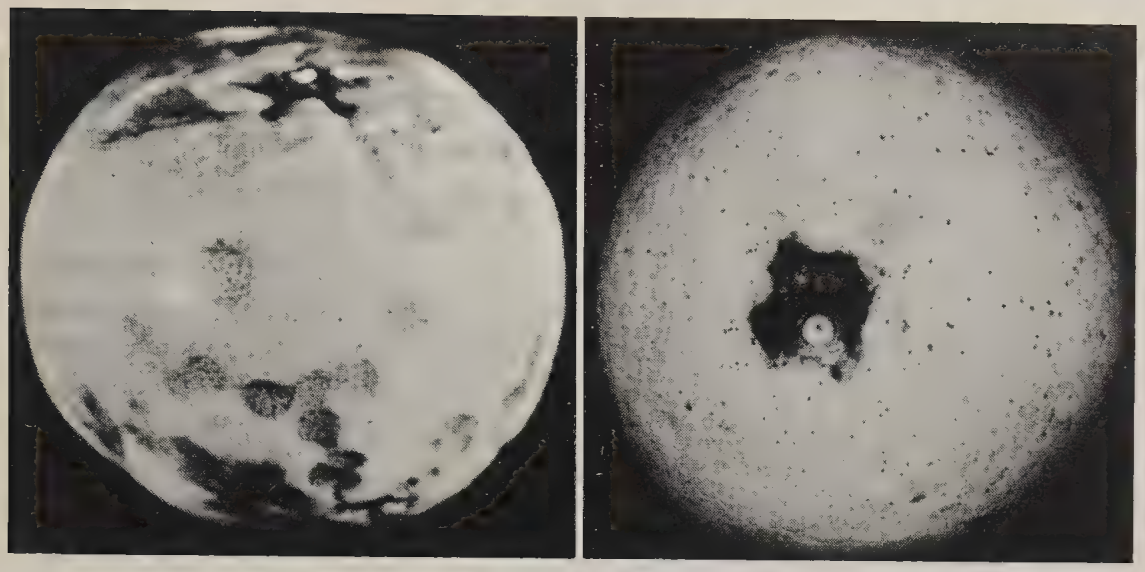


Fig. 133. Effects of various treatments and temperatures on the development of rind breakdown and decay of grapefruit after a 3-week simulated transit period and 2 weeks at 70° F. (21° C.). All crates contained 2 biphenyl pads. (From Chace *et al.*, 1966.)



Figs. 134 (left) and 135 (right). *Left:* Severe pitting symptoms on grapefruit held for 3 weeks at 40° F. (4.4° C.). *Right:* Aging on grapefruit stored for 3 weeks at 50° F. (10° C.), followed by 2 weeks at 70° F. (21° C.). (From Chace *et al.*, 1966.)

fruit stored at 32° F. (zero centigrade). No significant differences in the amounts of decay were observed in fruit stored at 40, 50, and 60° F. (4.4, 10, and 16° C.). It should be noted that most of the decay occurred during the 2-week holding period.

Since grapefruit rind is responsive to packing-house and pretransit treatments, it is important to determine the comparative effects of various measures that are used to protect the fruit against breakdown before it reaches the consumer. Chace *et al.* (1966) made such a study in determining the effects of washing, waxing, and temperature on the breakdown and decay of grapefruit stored under simulated export transit and holding conditions (figure 133). The fruit, washed or unwashed (but not waxed), developed excessive amounts of rind breakdown when held at 40° F. (4.4° C.). The waxed fruit (histograms *C* and *D*) held at 40° showed a marked reduction in rind breakdown. The application of wax to the fruit significantly reduced pitting, but did not eliminate it completely. The same treatment had a tendency to reduce the amount of decay. The waxed fruit stored at 50° F. (10° C.) had less rind breakdown than the washed samples, but the unwashed fruit showed the least injury. Fruit held at 60° F. (16° C.) had an exceedingly small increment of breakdown. In the 60° samples, the differences in the amounts of decay induced by the various treatments were very small.

The same authors submitted 2 photographs to illustrate the differences between the pitting and aging of grapefruit occurring under various storage and holding temperatures. As shown by figure 134, one of the most important low-temperature disorders of grapefruit is pitting, which consists of sunken areas in the rind at various degrees of intensity and with various amounts of

discoloration. The control of these low-temperature disorders is an important economic factor in the storage life of the fruit.

Aging or senescence (figure 135) comprises not only increasing weakness of the cells, but alterations in the structure of the rind that occur as a result of environmental conditions during storage. For example, water loss of the fruit during storage is often accompanied by physiological rind breakdown if the fruit is well aerated. Aging in grapefruit, and in fruits in general, is the developmental result of all the physiological processes that change with the passage of time. Aging of the tissue is a general biological process that continues, to varying degrees, throughout the entire life of the fruit. Senescence is the consequence of biochemical changes that occur in the cells of the various tissues, but not all the reactions occur in all the tissues at the same rate and at the same time. Fruit in the mature stage is essentially in a state of continuous physiological deterioration. An all-important aim of the fruit industry is to reduce the rate of these metabolic reactions, that cause deterioration, by regulating the environmental conditions under which the fruit is stored. It thus follows that the life span of postharvest fruits can be increased by judiciously regulating the temperature.

Grierson and Newhall (1955) observed an extraordinary rind breakdown on pink and red grapefruit for 3 consecutive seasons; it was first identified at a distinct disorder in 1954. They named it "sloughing" because the necrotic tissues remained moist and separated easily from the underlying healthy tissue, and the affected areas were confined to the flavedo and albedo of the peel. Sloughing originates on the surfaces of early pink and red grapefruit from 4 to 10 days after harvest, and has never been observed on attached fruit in the grove. This disorder appears to be partially associated with periods of high rainfall. It has been checked or controlled experimentally by storage at low temperatures (Grierson and Patrick, 1956). Sloughing is principally confined to Florida-grown grapefruit, but may occur in other areas.

Stem-End Rind Breakdown

COMMERCIAL VARIETIES of grapefruit sometimes appear to be susceptible to rind injuries that often resemble the peel defects occurring in oranges, commonly characterized as "stem-end rind breakdown" (SERB). These particular peel injuries are of considerable commercial importance in many varieties of oranges grown in humid climates, such as Florida and the West Indies (Grierson, 1965). Although it is not known to what degree the scientific data obtained on the SERB of oranges are applicable to grapefruit, it is important to present a characterization of these peel defects and to correlate the factors associated with the development of the disorder in oranges. The physiological data on rind breakdown with respect to relative humidity and air flow under postharvest storage conditions are probably not confined to the orange.

Hopkins and McCornack (1958) defined stem-end rind breakdown in oranges as a collapse of the peel tissue in the stem-end portion of the fruit, with the defects frequently extending several centimeters from the button. The affected tissues become hard and rough and, with the oxidation of the affected tissues on exposure to air, a brownish color develops which has given rise to the term "brown stem" (DeFossard, 1957). Their data indicate that this particular rind breakdown is a physiological deterioration which is strongly influenced by weather conditions, and especially water relations, during fruit growth, and in fruit under postharvest conditions. It should be emphasized that stem-end rind breakdown is a physiological disturbance of oranges, while stem-end rot is a pathological disease caused by certain species of fungi.

Hopkins and McCornack (1960) reported that Hamlin, Parson Brown, Pineapple, and Valencia orange varieties were susceptible to stem-end rind breakdown when subjected for 2 or more days to low relative humidity and high rates of air flow before being washed, waxed, and packed. When fruit was held under adverse postharvest conditions, rind breakdown (SERB) appeared 6 to 7 days after picking. They concluded, therefore, that stem-end rind breakdown in oranges is significantly accelerated during degreening by a high rate of air flow and low relative humidity, which factors cause excessive transpiration, as well as softening, of the fruit. Conversely, low air flow and high humidity during degreening will prevent stem-end rind breakdown in oranges.

If higher relative humidities are maintained, rind breakdown will not occur at fairly high rates of air flow.

Delay in the time of packing oranges after they were detached from the tree was highly associated with increase in stem-end rind breakdown and increase in fruit spoilage (Hopkins and McCornack, 1960). As a result of delayed handling, 34 per cent (mean value) of the fruit developed stem-end rind breakdown, while only 2 per cent developed rind breakdown in fruits that were washed, waxed, and packed the same morning they were picked. Delayed handling caused a significant increase in fruit decay, which extended from 17.2 per cent in promptly handled fruit to 36.7 per cent when handling was delayed. Stem-end rind breakdown was highly correlated with fruit decay, which indicated that the weak condition of the peel tissues served as a source of infection for pathogenic fungi.

In some preliminary experiments with both Marsh and Ruby Red grapefruit, Davis *et al.* (1963) reported that these varieties were not susceptible to rind breakdown as a result of delay in fruit handling or of time on the brushes. The samples of Marsh and Ruby Red were held for 4 and 5 days, respectively, at 70° F. (21° C.) and 85 per cent relative humidity, and were subsequently brushed for 3 minutes. The fruit was inspected a week after treatment, and was found to be free of rind breakdown.

Wrappers and Waxes

SOME EARLY STUDIES on the effects of 20 different kinds of wrappers on the keeping quality of grapefruit stored at different temperatures were made by Stahl and Fifield (1936). They presented their data on both oranges and grapefruit in 26 tables. They stated that for grapefruit, the moistureproof wrappers (aluminum foils, and moistureproof grades of cellophane, Sylphrap and Kodapak) were superior to all others in reducing weight loss and preserving the normal general appearance of the fruit. Next in order came wet-waxed and paper parchment wrappers, semi-moistureproof grades of cellophane, and plain tissue wraps. Temperature, and not wrappers, was found to be the controlling factor in the pitting of grapefruit. Tissue wraps had little effect.

The efficiency of the wrappers in preventing decay varied at different temperatures, from 32 to 80° F. (zero to 27° C.); i.e., no one wrapper was consistently good or consistently bad at all temperatures. A given wrapper might be good at one, medium at another, and bad at a third temperature. Temperature, and not the type of wrapper, was also the controlling factor in the retardation and prevention of decay—37.5° F. (3° C.) was found to be the best. Loss in fruit weight, as would be expected, was in direct relation to the temperature: the lower the temperature, the smaller the loss. Stahl and Fifield found that grapefruit was kept in good condition for 3 to 4 months in moistureproof wrappers, for 2 to 3 months in semi-moistureproof wrappers, while fruit wrapped in plain tissue lasted only from 6 weeks to 2 months. The box liner was found to be as efficient as the individual wrapper in all respects, except in preventing "nesting" (masses of decayed fruit) . The combination tissue wrap and moistureproof box lining proved to be most satisfactory. One-sixth as much material was used in box lining as when individual wrappers were used. Fruit wrapped in moistureproof wrappers and held at 37.5° F. kept best after removal from cold storage to store temperature.

Stahl and Vaughn (1942) determined the relative loss in weight of Duncan grapefruit wrapped in 40-gauge pliofilm and stored at 70 and 42° F. (21 and 5.5° C.) (figure 136). The nonwrapped control fruit showed large weight losses at both temperatures. After 3 weeks, the control fruit stored at 70° was unsalable as the result of excessive loss of moisture. During this storage period, the total weight of the wrapped fruit decreased less than 1 per cent. In addition to the reduction in weight loss, the wrapped fruit contained much less rind pitting, with only a slight amount appearing after 6 weeks of storage. In the nonwrapped fruit, general pitting occurred after 3 to 4 weeks' storage, and it was more severe at 42° than at 70°.

Earlier than the work done by Stahl and Fifield (1936), Friend and Bach (1932) reported that waxed wrappers kept the fruit in better condition than was the case with ordinary wrappers after it was removed from storage.

Van der Plank and Rattray (1938*b*) found that waxed crystalline wrappers reduced losses in weight and by pitting, but increased loss caused by molds.

Martin *et al.* (1939), in Arizona, reported that waxed fruit lost only 52 to 74 per cent as much moisture as nonwaxed fruit when stored at 60° F. (16° C.) and 88 per cent relative humidity. Tests were made once a month, December through May.

Rose *et al.* (1951), in reviewing the reported effects of wrappers on the keeping qualities of stored grapefruit, stated that most of such studies had

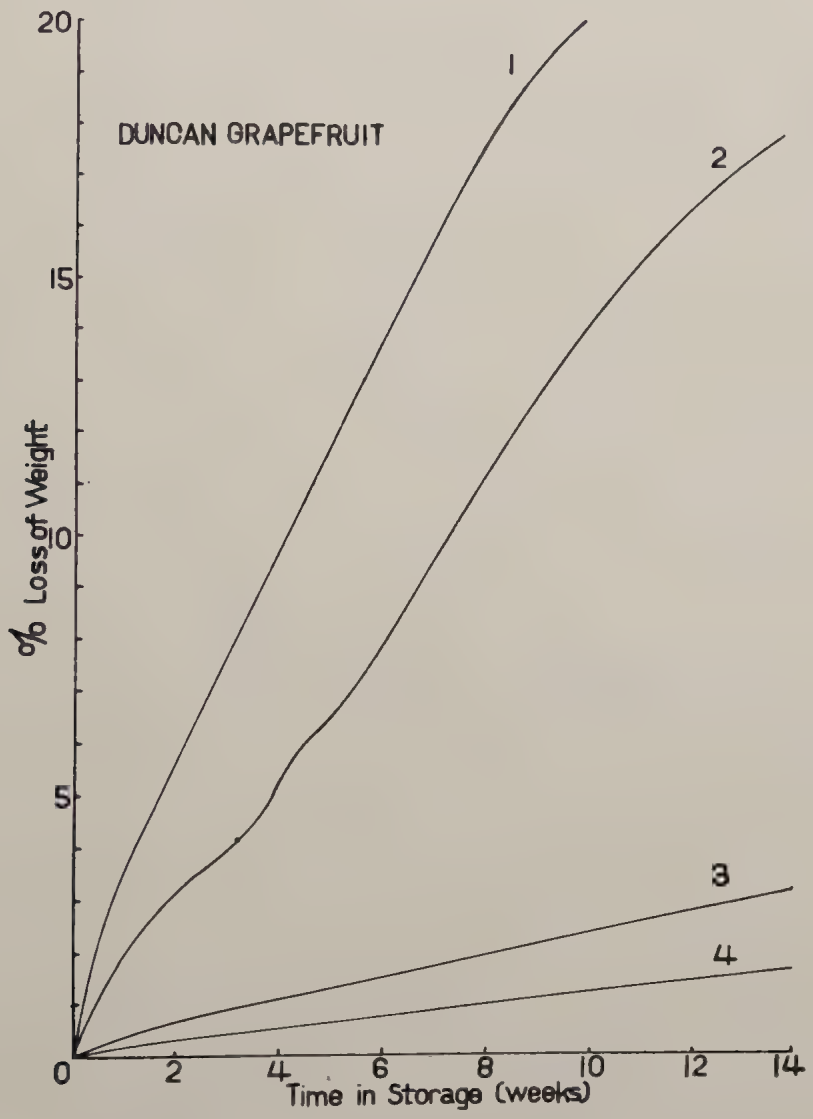


Fig. 136. Percentages of weight loss of Duncan grapefruit during 14 weeks' storage, when wrapped in 40-gauge pliofilm and when nonwrapped. At 70° F. (21° C.), (1) indicates nonwrapped and (3), wrapped fruit. At 42° F. (5.5° C.), (2) indicates nonwrapped and (4), wrapped fruit. (From Stahl and Vaughn, 1942.)

been confined to oranges and lemons. They drew the general conclusion that wraps and waxing contributed such advantages that under normal storage and marketing conditions, they should not be ignored. They added that although many citrus-producing countries were shipping fresh citrus fruits in cartons and bags without wrappers, some others were continuing to export the fruit in wraps impregnated with biphenyl.

Martin *et al.* (1939) also studied the effects of time of picking and waxing on loss of weight in storage (figure 137). In Arizona, it was standard practice

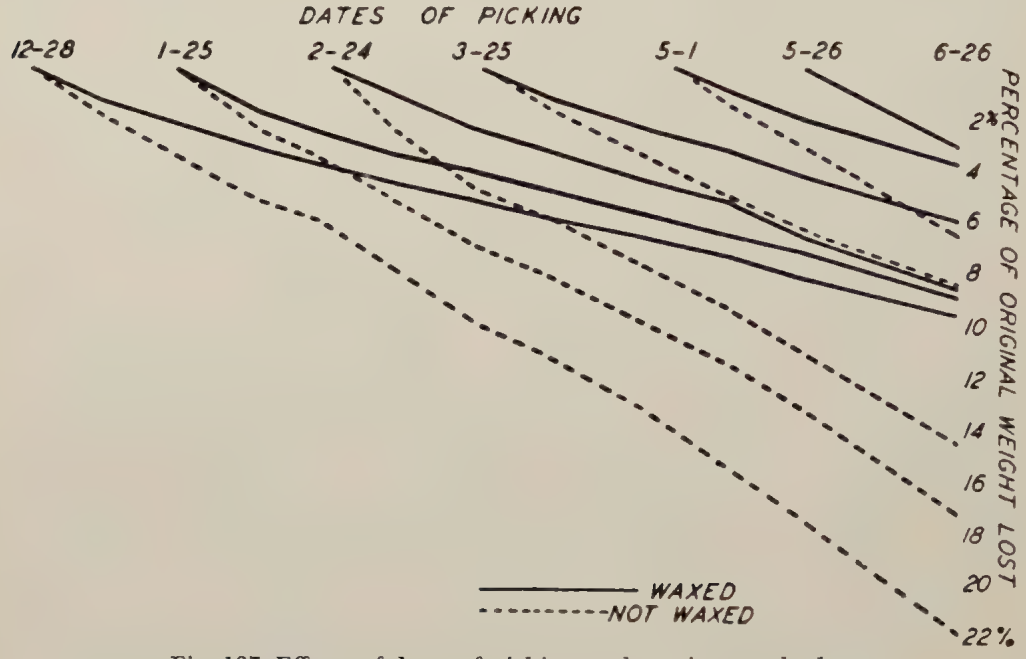


Fig. 137. Effects of dates of picking and waxing on the loss of weight of stored grapefruit. (From Martin *et al.*, 1939.)

to wax grapefruit before storage. The material is applied to the fruit as a water-wax emulsion which, on drying, leaves a thin film of wax covering the surface. The data in the figure show clearly that the wax treatment before storage was beneficial in prolonging the life of the fruit by reducing shrinkage. The waxed fruit also lost far less weight than the nonwaxed fruit after removal from storage.

The same authors also determined the effect of waxing on the concentration of soluble solids in the juice of stored grapefruit. The fruit samples were taken from 3 of the main producing areas near Phoenix, Arizona. The fruit was washed and tested on January 24, 1939, then half was wax-treated, and all was placed under regular storage conditions. Further tests were made at approximately monthly intervals, with the results shown in table 171. Martin and his co-workers stated that the relatively high values in the nonwaxed fruit could probably be attributed to the shrinkage of the fruit and hence the higher concentrations of juice.

Vines *et al.* (1968) determined the relative percentages of oxygen and carbon dioxide in the internal atmosphere of Marsh grapefruit as influenced by the thickness of the wax coating on the surface (figure 138). The respiration rate of grapefruit did not change significantly with 3 coats of wax, but the internal oxygen decreased simultaneously with an increase in carbon dioxide. The gaseous composition within the tissues was somewhat associated with the metabolic reactions of the fruit, and consequently, changes occurring in the internal gaseous concentration were correlated not only with changes in the rate, but in the nature of the biochemical reactions taking place. In these experiments, the permeability of the peel or epidermis to oxygen and carbon dioxide was unquestionably decreased by the 3 applications of wax to the surface. These results were similar to the data reported by Eaks and Ludi (1960) and Ben-Yehoshua (1967) for Washington Navel and Shamouti oranges, respectively.

Moisture loss begins in grapefruit as soon as it is detached from the tree. The loss of weight during storage is chiefly caused by this water loss. The rate at which water is lost is a function of the chemical nature of the surface area of the fruit and the characteristics of the environment; i.e., relative humidity and temperature. Water loss from fruit is related to the differences in vapor pressure in the fruit and in the surrounding atmosphere. It thus follows that the vapor-pressure deficit is the difference between the actual water-vapor pressure and that needed to saturate the air at the same temperature. Since the amount of water in the atmosphere (vapor pressure) is a function of temperature, the loss of weight by grapefruit in storage is least at lower temperatures with higher humidities.

Wells (1962) graphically illustrated the weight loss in grapefruit at 60° F. (16° C.) by plotting the loss in weight per day against the relative humidities of the storage atmosphere and the corresponding vapor-pressure deficit values

TABLE 171

EFFECTS OF WAXING ON THE TOTAL SOLUBLE SOLIDS OF STORED GRAPEFRUIT.*

Dates tested (1939)	Soluble solids (per cent)	
	Fruit waxed	Fruit not waxed
January 24	12.50	12.50
February 22	12.93	13.23
March 21	12.82	13.22
April 20	12.89	13.32
June 1	12.88	13.17
June 29	12.63	12.68

*From Martin *et al.* (1939).

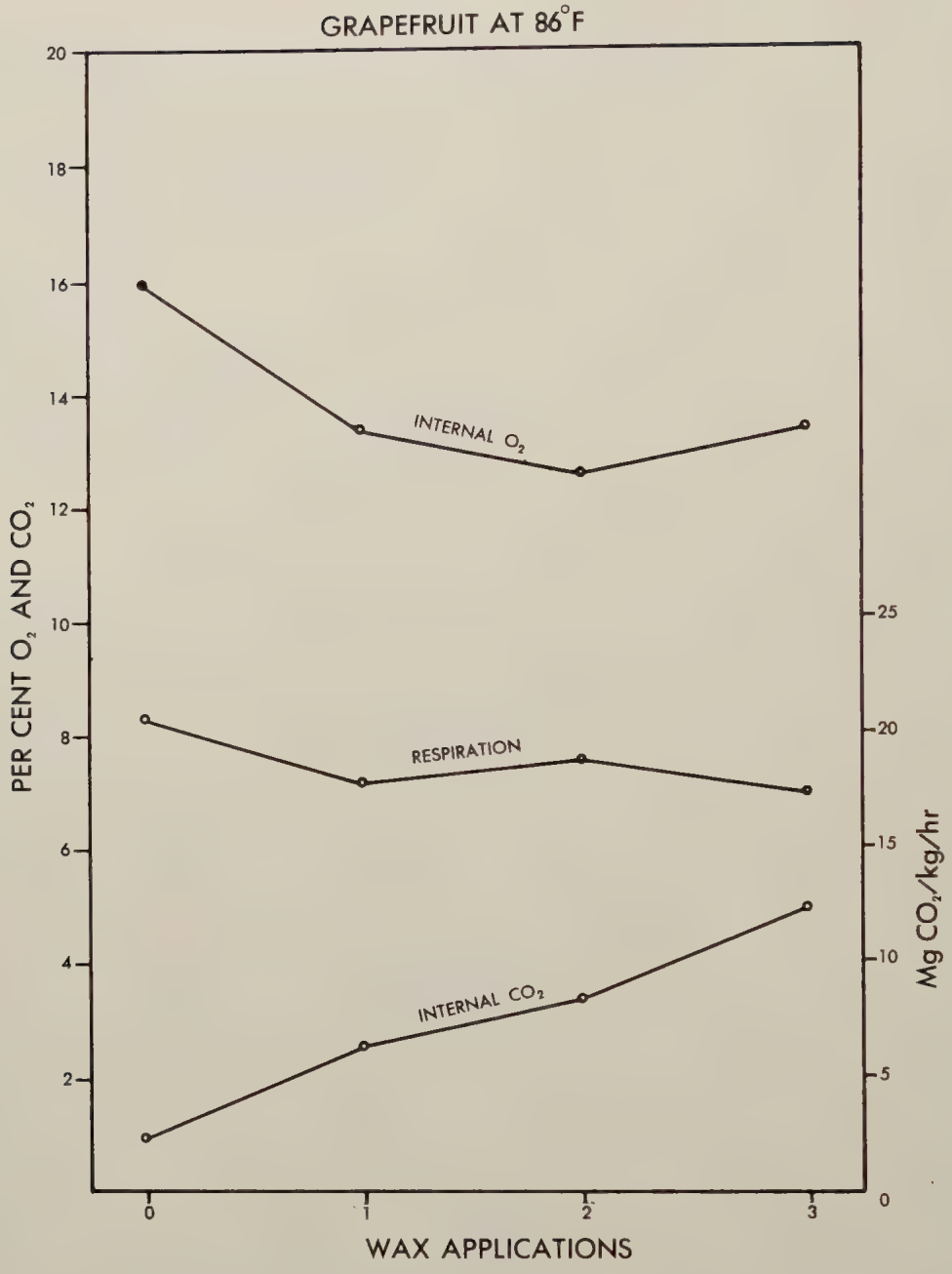


Fig. 138. Analyses of internal oxygen and carbon dioxide as percentages with increasing wax thickness in Marsh grapefruit, stored at 86° F. (30° C.). The respiration rates were recorded as milligrams of carbon dioxide per kilogram/hour, using 3 separate fruits per treatment, with 4 analyses daily for 1 week. (From Vines *et al.*, 1968.)

(figure 139). Between relative humidities of 91 and 80 per cent, the weight loss (mostly water) varied from 77 to 135 milligrams per 100 grams per day. The rate of weight loss in grapefruit was considerably less than in oranges and lemons. Since the loss of weight varied inversely with fruit size, it appears that for a given weight of fruit, the small fruit would expose a greater external surface area to the atmosphere. Although no penetration experiments were performed on grapefruit, the data obtained on oranges should be equally applicable. Softening of oranges, as measured by their compressibility in millimeters, was found to be highly associated with weight loss as well as with vapor-pressure deficit.

Respiration

A PROCESS OF COMBUSTION of stored food materials in which oxygen is absorbed and oxidative products released, largely in the form of carbon dioxide, is termed *respiration*. This process results in the liberation of energy and a reduction in the total dry weight. These products—energy, weight, and carbon dioxide—are measurable as indicative of the rate and amount of respiration. The chemical aspects of the respiration process may be represented empirically by the equation:

$$C_6H_{12}O_6 + 6O_2 \text{ (enzymes)} \rightarrow 6CO_2 + 6H_2O + 673 \text{ kg-cal.}$$

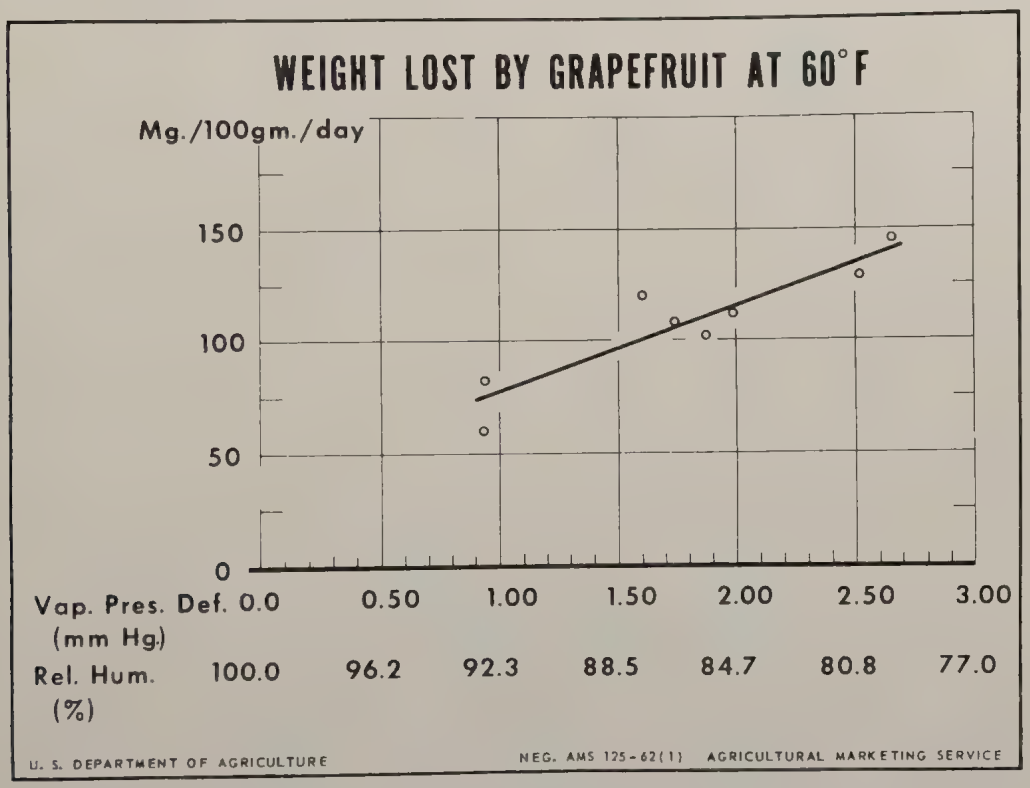


Fig. 139. Weight lost by grapefruit at 60° F. (16° C.) as a function of vapor-pressure deficit and relative humidity. (From Wells, 1962.)

The heat liberated (673 kg-cal) is based on the assumption that glucose is the hexose oxidized. The amount of energy released by the oxidation of other hexose sugars deviates only slightly from that of glucose. This empirical equation does not indicate in any way the intermediate steps in the process of respiration. The equation merely states that 6 moles of oxygen are required to oxidize 1 mole of a hexose to 6 moles of carbon dioxide and water with the release of 673 kg-cal of energy.

It should be noted that most reactions at room temperature double or triple their rates for a 10° C. rise in temperature (Van't Hoff's law). This fundamental generalization finds important application in biological reactions. Reactions which vary in rate by a factor of 2 or 3 over a temperature difference of 10° C. (the equivalent of 18 degrees F.) usually have their energy of activation (Eact) in the order of 10–20 kg. cal./mole. The temperature coefficient (Q_{10}) is the term applied to the factor by which the rate constants differ over a temperature interval of 10° C.

As with chemical reactions, the respiration rates of fruits are associated within limits with temperature. At lower temperatures, the rates increase exponentially; at higher ones, they decrease with time. Depending on fruit condition, higher temperatures are more effective in increasing the rate of physiological breakdown than the respiration rate.

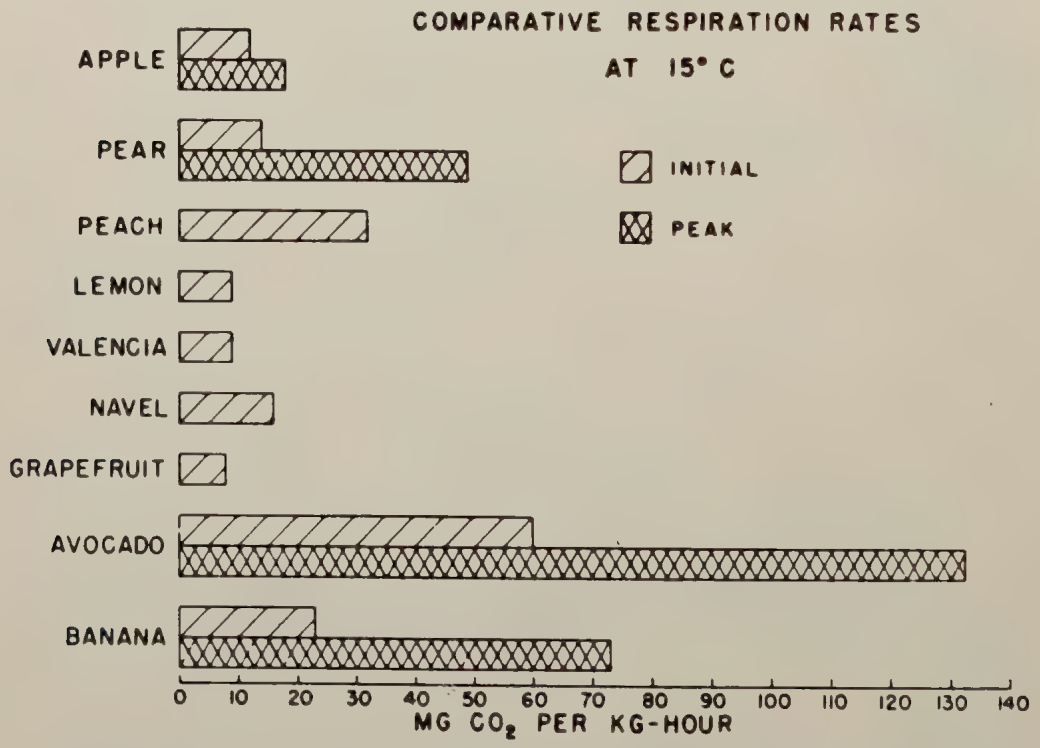


Fig. 140. Comparative respiratory rates for several fruits at 15° C. (59° F.). "Initial" refers to values obtained immediately after harvesting. "Peak" means respiratory activity at the climacteric maximum. No climacteric peaks were observed in citrus. (From Biale, 1954.)

Biale (1954), in his research on the metabolic rate of grapefruit, compared the respiration rates of citrus fruits with those of deciduous and other subtropical fruits (figure 140). The initial values on the bar diagrams show the respiration rates of the various fruit samples immediately after removal from the tree. The peak values represent the respiration rates that occurred at the maximum height of the curve, commonly referred to as the "climacteric." No climacteric peaks were observed in the respiration curves of grapefruit and other citrus types. Grapefruit had the lowest rate of respiration noted for

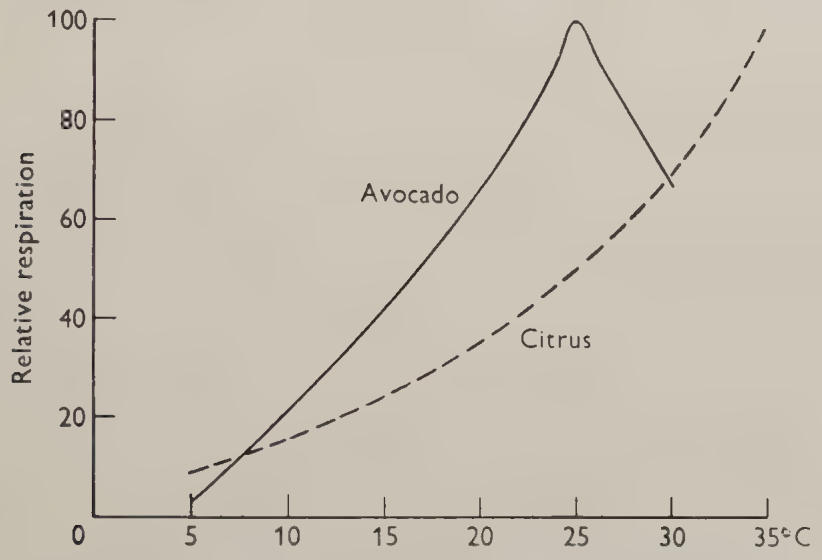


Fig. 141. The effects of temperature on the respiratory rates of avocado and citrus fruits. (From Biale and Young, 1962.)

citrus fruits. The low rates of respiration of citrus fruits and the unobservable climacterics are associated with the slow rates of chemical reaction that occur in these fruits during growth and maturation.

Since most chemical reactions are accelerated at high temperatures, the rates of respiration and ripening of citrus fruits are also affected by changes in temperature, humidity, and other environmental factors. With respect to temperature, the rate of respiration of fruit is related to the temperature within a range of 5 to 30° C. (41 to 86° F.). At 30°, the climacteric rise in respiration of avocados is suppressed, and abnormal softening usually occurs, but at temperatures from 10 to 25° C. (50 to 77° F.), avocados exhibit a definite climacteric with normal ripening of the fruit. Avocados stored at 5° C. for 17 days showed no climacteric rise in respiration, but the fruit did exhibit chilling injury (Biale and Young, 1962). Their data in figure 141 illustrate the relative respiration rates between the climacteric (avocado) and nonclimacteric (citrus) fruits at various temperatures. The respiration rates of avocados were more highly responsive to temperature than those of citrus. In the avocado, the maximum respiration preceded the onset of softening

by 1 to 3 days. In other words, the climacteric class of fruits had a different response to temperature from that of the nonclimacteric class.

In the normal ripening of fruits, the temperature ranges involved are related to the numerous enzymatic reactions within the fruit. For a given enzyme, the optimum temperature is the one at which the activity of the enzyme is at a maximum, and it varies with the concentration of the enzyme, time of the reaction, and the pH of the reaction medium. Owing to the multiple-enzyme systems that are present in fruits, the rates of reaction of the various enzymes in the tissues would be different, and consequently, their optimum activities would require different physical and chemical characteristics.

Biale and Young (1962) also stressed the importance of controlling the temperature and gases in regulating the respiration and ripening of harvested fruits. Cold-sensitive fruits that had been subjected to chilling temperatures for a required period did not exhibit the climacteric pattern of respiration.

Biale (1941) observed no climacteric rise in the respiration of avocado fruits stored at 5° C. (41° F.) over a period of 5 weeks. When these samples were transferred to 15° C. (59° F.) after 2 or 3 weeks at 5°, the typical upsurge in respiration occurred. He noted, however, that prolonged storage of avocados at 5° caused a decreased rate of CO_2 production after removal of the samples to 15°. Apparently, chilling injury occurred with the alteration of the climacteric pattern. The lowest temperature at which a rise of respiration was observed in the Fuerte avocado was 7.5° C. (45° F.) (Biale, 1946).

In contrast to climacteric fruit, such as the avocado, citrus fruits are typical examples of nonclimacteric fruit when viewed in relation to chemical changes that occur after harvest. In his investigations on postharvest changes in oranges, Biale (1960*a*) observed that oranges and grapefruit attained desirable composition and matured on the tree, not after harvest, as do the climacteric fruits.

The early and midseason oranges and grapefruit undergo the same physiological trends after harvest as they do on the tree, except that the rates of change are slower, especially when the fruits are placed in storage at correct temperatures. As oranges grow to full size and maturity, the total soluble solids increase and the total acidity decreases. As these changes gradually occur over a prolonged growing season, oranges can be stored on the tree for various lengths of time after they reach the ripe stage, depending, of course, upon varietal differences. It has been well established that Valencias keep better than Washington Navel and Shamouti oranges, both on and off the tree. Fruits that do not have a pronounced climacteric rise contain no starch, and usually complete the maturation process while attached to the parent plant. Changes that occur in storage, specifically in grapefruit, are discussed elsewhere in this chapter.

Aharoni (1968), in his studies on the physiological changes occurring in oranges stored in a modified atmosphere, conducted experiments in which the respiration rates of oranges and grapefruit were determined on detached fruits at different stages of development (figure 142). The grapefruit samples were harvested at 6 different periods, from May 22 to December 14 (*A* to *F*), and the average fruit weight of the samples varied in size from 13.5 grams to 392 grams during this period. An inspection of the curves shows that an initial decrease in the rate of respiration usually preceded the upsurge of

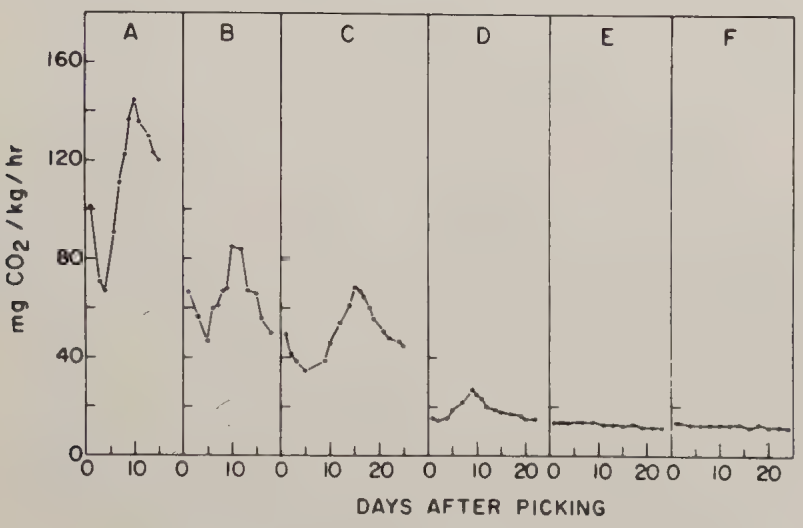


Fig. 142. Respiratory rates of Marsh Seedless grapefruit at 20° C. (68° F.), harvested at 6 different stages of development. (From Aharoni, 1968.)

carbon dioxide production. The maximum peaks were observed in the youngest fruits, and the rates of respiration, as shown by the lower heights of the peaks, were reduced with an increase of fruit growth and development. This gradual decrease in respiratory activity with seasonal development of the fruit was not followed by an upsurge of carbon dioxide production. The disappearance of the respiratory peaks was associated with varietal differences in maturity dates. In early-maturing grapefruit, the rise in respiration was not present in late October and early November fruit samples (stages *E* and *F*); in the later-maturing varieties (Shamouti and Valencia), the respiratory upsurge did not disappear until mid-December.

The respiratory patterns of young and unripe grapefruit, when expressed in terms of carbon dioxide liberation, were similar to the curves produced by climacteric fruits on ripening. The time course of respiration showed a decrease in carbon dioxide shortly after harvest, with a subsequent upsurge to a peak, followed by another decrease. It was further shown that the increase in respiration was accompanied by characteristics common to climacteric fruits, such as an increase in ethylene production, changes in color of peel

and pulp, and abscission of the stem ends. Aharoni stated that his data supported the conclusions of Trout *et al.* (1938, 1960), who reported that oranges exhibited a climacteric peak, particularly if the fruit was harvested before the commercial ripening stage.

Although no ethylene data were submitted for grapefruit, Aharoni (1968), in figure 143, determined the respiration and ethylene production of 3 vari-

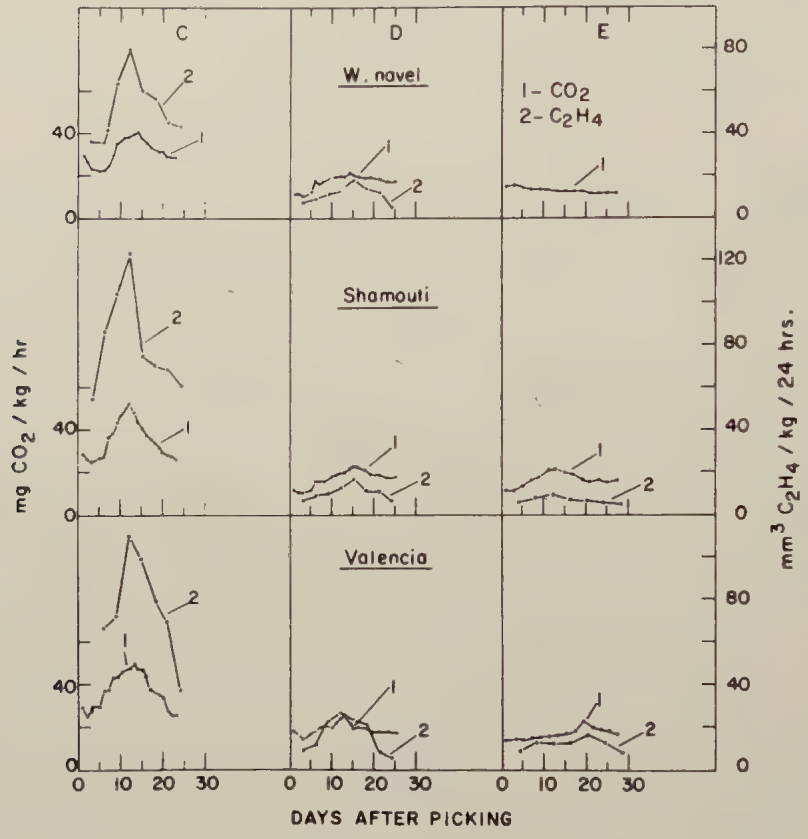


Fig. 143. Respiration and ethylene production of Washington Navel, Shamouti, and Valencia oranges at 15° C. (59° F.), harvested at 3 different stages of development. (From Aharoni, 1968.)

eties of oranges harvested at 3 different stages of development (*C, D, E*), July 23, September 6, and November 1. In the immature fruits, the curves showing the rates of ethylene production run parallel to those of carbon dioxide liberation, both types of curves reaching their peaks at the same time, and decreasing correspondingly. The younger fruit produced the greater quantity of ethylene, and the amounts present decreased with fruit growth and maturity. In the ethylene production curves, the highest peaks occurred in the youngest fruits. No ethylene was present in Washington Navel oranges harvested in stage *E*, while in the Shamouti and Valencia varieties, no ethylene production was observed in stage *F*. (*See figure 142 on page 451.*)

Eaks (1970), in his studies on the respiratory patterns of citrus fruits of different ages, reported that small, immature grapefruit and oranges (both Valencia and Washington Navel) not only showed an increased rate of respiration a few days after harvest, but at the same time produced effective amounts of ethylene (figure 144). However, lemon fruits showed no comparable respiratory rise at any stage of development. Data were presented showing that cutting the pedicel caused no immediate change in the respiratory rate of orange fruits, which indicated that detached citrus fruits would give a reliable index to the respiratory rate of fruit on the tree. The data in the figure show that the initial respiratory rates of citrus fruits (grapefruit, oranges, and lemons) decreased as the fruit increased in weight. However, fruits harvested after September 4 showed only slight decreases in initial rates as they increased in size.

The respiratory rate increase which began 3 to 4 days after harvest for grapefruit and Valencia and Washington Navel oranges (figure 144) resembled the climacteric in other mature fruit, but was not considered to be a true climacteric, primarily because citrus fruits can be restimulated with ethylene. The peak respiratory rate and the ratio of the peak rate to the prerise minimum decreased with an increase in fruit weight. Ethylene production in physiologically significant amounts occurred during the respiratory rise with grapefruit and oranges, but decreased as the fruits matured and increased in size. No respiratory rises and no detectable amounts of ethylene were produced by larger, mature fruits. The influence of this endogenously produced ethylene was similar to that of exogenously applied ethylene in producing a rind color, change from green to yellow, and causing abscission of buttons. No explanation was given as to the low responses of lemons.

Eaks (1970) concluded that citrus fruits should be considered as nonclimacteric, based on the evidence of their respiratory response to ethylene and the lack of physical and chemical changes normally accompanying the climacteric, and that the respiratory rise displayed by small, immature grapefruit and oranges was the result of an unexplained ethylene production.

When mature grapefruit was subjected to a variety of postharvest conditions, Vines *et al.* (1968) reported that certain anomalies were observed to be associated with the presence of ethylene in the internal atmosphere. In a series of controlled freezing experiments, they found that not only was respiration increased as a function of time, but at the maximum level of respiration, ethylene was detected at 6 ppm. Grapefruit dropped to a hard surface from a height of 4 to 6 feet (122 to 183 centimeters) showed an increase in ethylene in the internal atmosphere. These and other data led the authors to suggest that the evolution of physiological amounts of ethylene by citrus fruits would be a symptom of stress. (See also, figure 83 of chapter 4, with its accompanying text—pages 285 and 287.)

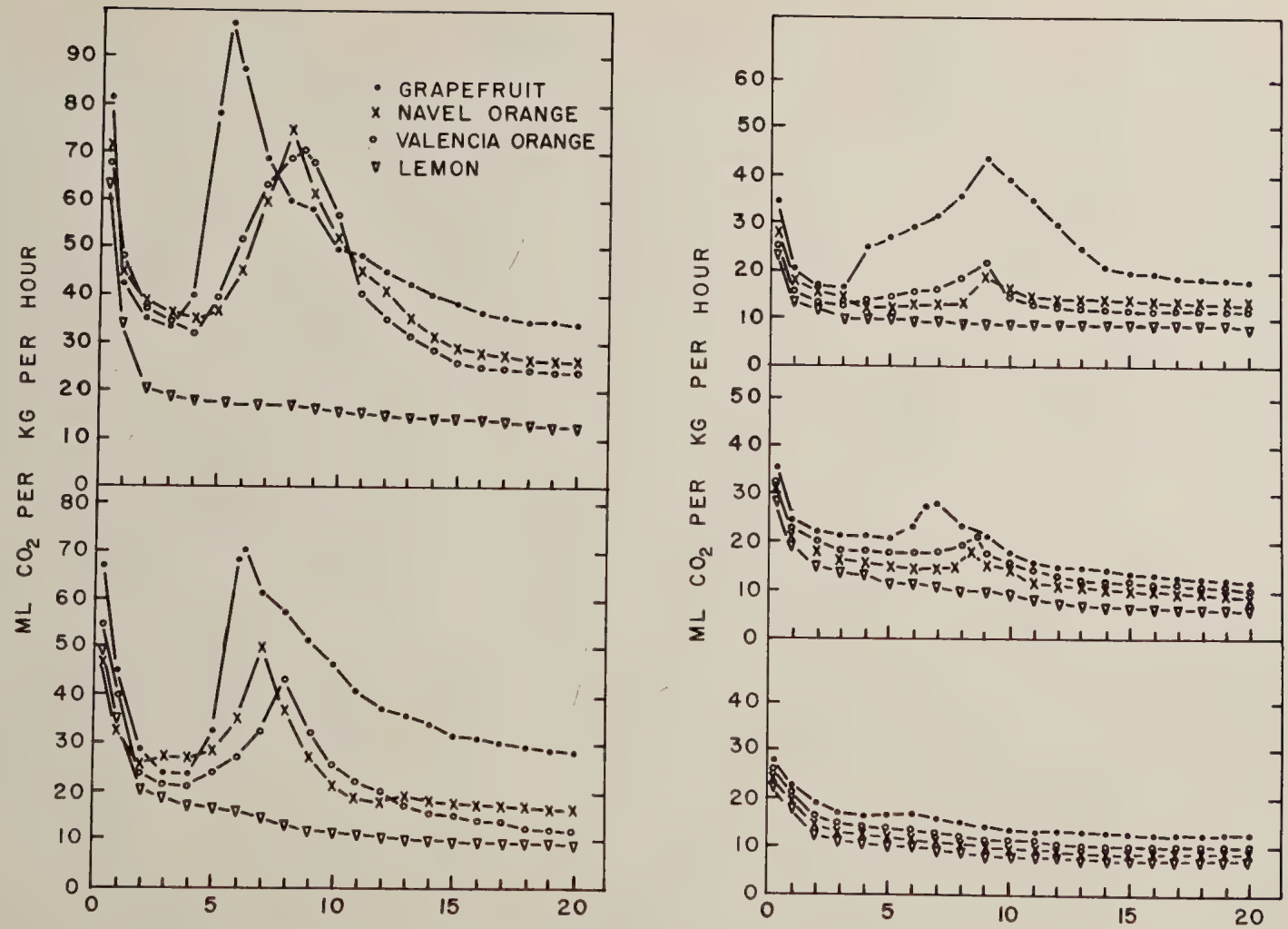


Fig. 144. Respiratory rates of 4 citrus fruits at 5 picking dates. Fruit weights are stated in grams, in the same order as named at upper left. Figures at bottom represent number of days after harvest, *Upper left:* June 13; 15, 17, 15, 14 grams. *Bottom left:* July 1; 31, 25, 25, 24 grams. *Top right:* July 22; 40, 34, 33, 31 grams. *Center right:* August 16; 62, 70, 50, 37 grams. *Bottom right:* September 4; 120, 87, 64, 61 grams. (From Eaks, 1970.)

Langworthy and Barott (1922) were among the first to determine the rate of respiration of stored grapefruit. Each sample of 123 kilograms (about 272 pounds) was placed in a calorimeter, which in turn was placed in a sealed room at 78° F. (25.5° C.) for 8 days. Determinations were made at intervals of about 24 hours. The tests were conducted in Washington, D.C., on fruit sent from Puerto Rico. They obtained the following results: heat eliminated varied from 1.7 to 2.6 calories per hour; carbon dioxide eliminated varied from 1.8 to 2.4 grams per hour; water given off was quite constant at 23.8

TABLE 172

THE RESPIRATION RATES AND RESPIRATORY RATIOS OF SEEDLING GRAPEFRUIT IN RELATION TO TEMPERATURES.*

Temperatures (F. and C.)	CO_2 (mg per kg-hr.)	O_2 (cc per kg-hr.)	Ratios of CO_2 to O_2
32 (zero)	2.07	1.38	0.77
40 (4.4)	4.86	2.23	1.13
50 (10.0)	6.92	2.80	1.25
60 (16.0)	12.60	5.35	1.19
70 (21.0)	16.00	6.48	1.24
80 (27.0)	19.00	6.64	1.45
90 (32.2)	27.50	8.80	1.60
100 (38.0)	51.40	13.20	1.96
110 (43.3)	81.60	26.00	1.59

*Adapted from Haller *et al.* (1931).

to 25.6 grams per hour; while the oxygen absorbed also showed variation—0.7 to 3.1 grams per hour.

Haller *et al.* (1931) determined the effects of temperature on the rates of respiration of Florida seedling grapefruit (table 172). The respiratory ratios increased with increases in temperature. When complete oxidation of hexose occurred, the ratio was equal to 1. The complete oxidation of citric or malic acid yielded a ratio of 1.33. Ratios higher than 1.33 were indicative of intramolecular respiration. Apparently, these grapefruit samples had considerable intramolecular respiration at the higher temperatures. The authors found the heat of respiration to be relatively low. At 32° F. it was 455, at 50° it was 1,518, and at 60° it was 3,520 btu per ton per day (zero, 10, and 16° C.). At 32° the heat of respiration of the fruit they used would melt only 3.2 pounds of ice per ton of fruit per day.[2]

Eaks (1965), in studying the physiological responses of citrus fruits to chilling injury, determined the effects of continuous and intermittent chilling exposures to zero and 10° C. (32 and 50° F.) on the respiratory rates of oranges and lemons. He reported that the rates at 20° C. (68° F.) in fruits subjected

[2] One pound equals 0.454 kilogram. One ton equals 2,000 pounds.

to continuous chilling exposures showed a greater increase as the time of exposure increased. Fruits exposed to zero centigrade had a greater stimulation than those exposed to 10°. In experiments where the fruit was exposed intermittently to zero and 10° C., the maximum respiratory rates at 20° decreased as the exposure period decreased. The data presented strongly indicate that a cumulative time–temperature relationship was involved between the chilling exposure and the rate of production of carbon dioxide at nonchilling temperatures. The 2 classes of fruit, those that were susceptible and those that were nonsusceptible to chilling temperatures, had a fundamental difference in the physiology and biochemistry of their respiratory mechanisms. Furthermore, it was observed that the maximum respiratory rate attained 8 to 10 hours after the fruit was transferred to 20° C. exceeded that observed for nonchilled fruit.

Vines *et al.* (1968), in experiments somewhat similar to those on oranges and lemons, determined the respiratory rates of Marsh grapefruit that had been subjected to –8° F. (–22° C.) for various periods up to 300 minutes (figure 145). During the 14-day respiration period, the carbon dioxide production of these fruits increased with the time of exposure to the freezing temperature.

No attempt was made to allocate the increased rate of carbon dioxide evolution between that caused by chilling injury at zero Fahrenheit (–18° C.) and above, and that caused by freeze damage. Extraordinarily, some grape-

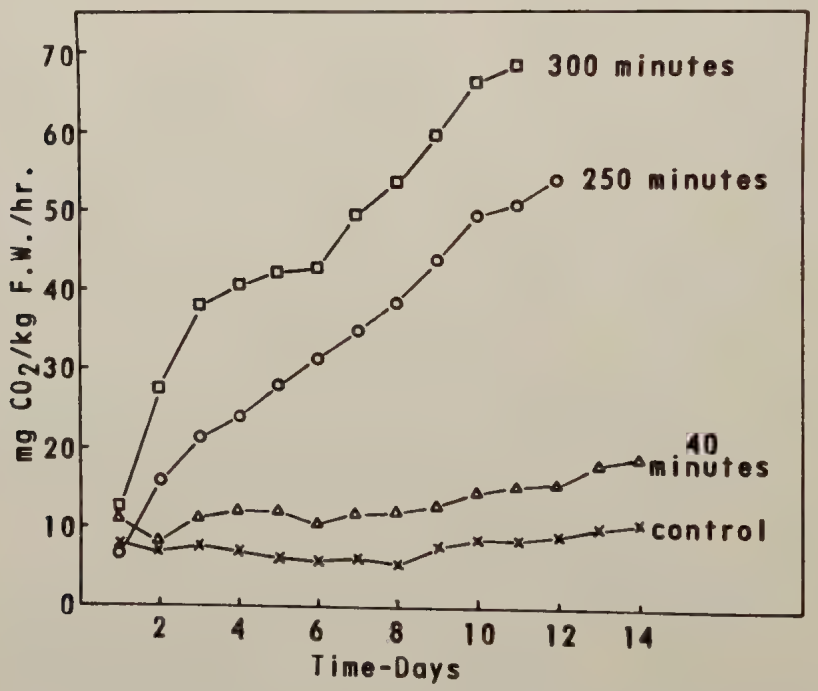


Fig. 145. Respiratory rates of Marsh grapefruit during a 14-day period after subjection to –8° F. (–22° C.) in each case. (From Vines *et al.*, 1968.)

fruit showed no increase in respiration, and produced no internal ethylene; the fruit retained good appearance and flavor for 30 days after the treatment.

Oxygen and Carbon Dioxide Tension in Storage Atmospheres. Stahl and Cain (1937, 1939) stated that both taste and texture of Florida-grown grapefruit were adversely affected by high concentrations of oxygen and carbon dioxide in the surrounding atmosphere. However, forced-air circulation in the storage room was found to be harmful to the texture of the fruit and hence to the length of its storage life. Grapefruit stored at 37° F. (2.8° C.) in still air containing about 6 per cent carbon dioxide and 12 per cent oxygen, and with high relative humidity, was kept in good condition for 4 months. The authors noted that respiratory gases should not be permitted to accumulate.

Biale and Young (1947, 1962) made extensive studies of the effects of modified atmospheres on the respiration and ripening of harvested fruits, and arrived at the following general conclusions: Fruits grow, mature, and ripen in normal atmosphere (composition by volume, 0.03 per cent carbon dioxide, 21 per cent oxygen, 78 per cent nitrogen, small concentrations of inert gases, and various amounts of water vapor). Nitrogen, the highest component of the air, appears to be physiologically inert, since it can be replaced in this experimental system by hydrogen or helium without changing fruit behavior. When fruits are removed from the tree, it is highly important that their physical and chemical reactions should be determined and controlled when they are placed under conditions of storage and transportation. The storage life of citrus fruits may be prolonged experimentally by regulating the environmental conditions. The ripening process of citrus fruits is significantly different from that of the climacteric class in that their rates of respiration (grapefruit, orange, and lemon) are comparatively low, and are characterized by a gradual decline with time, with no sudden upsurge in carbon dioxide under conditions of variable temperature. Consequently, the nature and rates of the biochemical reactions of the ripening process can be markedly influenced by modifying the environmental conditions of stored fruits.

The influence of oxygen concentration on the rates of respiration of both avocados and citrus fruits is shown in figure 146. The respiration curve for the Fuerte avocado shows the values for the climacteric peak plotted as a function of oxygen concentration. At low concentrations, the steady-state values were used. The rates of carbon dioxide production dropped markedly at partial pressures of oxygen lower than in the atmosphere, with the resultant effect of delaying the onset of the climacteric rise. At an oxygen concentration higher than that in air, the rate of respiration gradually increased to an oxygen concentration of approximately 35 per cent, at which point the curve leveled off with no further increase in respiration at the higher percentages of oxygen. In the avocado, the oxygen absorption followed closely the carbon dioxide evolution, with the respiratory quotients slightly less than

unity. The variations in respiratory quotients at low oxygen concentrations suggested no change other than the normal respiratory process.

The respiratory curve of citrus fruits at various concentrations of oxygen was significantly different from that of the avocado (figure 146). In citrus fruits, there occurred a critical oxygen concentration between 2.5 and 5 per cent at which the carbon dioxide evolution was at a minimum. The respiration of citrus fruits increased with a corresponding increase in oxygen tension between 5 and 100 per cent. The respiratory quotient was at a minimum

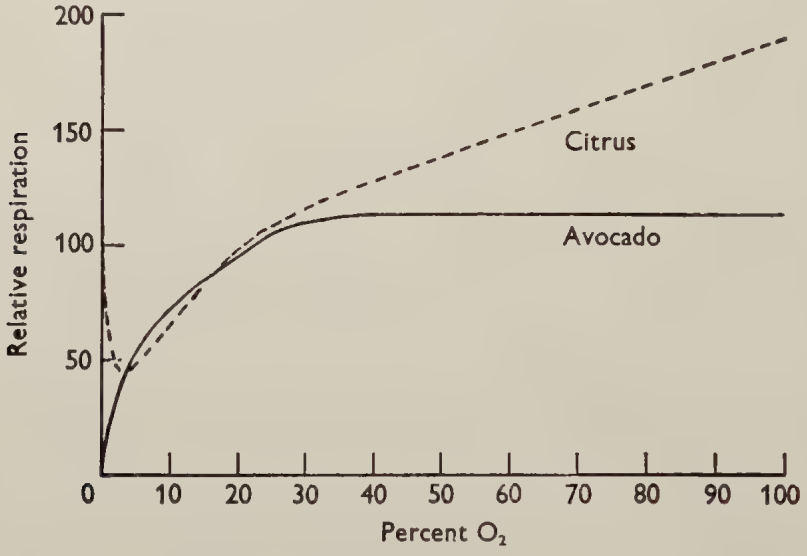


Fig. 146. Respiratory rates of avocado and citrus fruits as functions of oxygen concentration of the storage atmosphere. (From Biale and Young, 1962.)

at 5 per cent oxygen concentration. At oxygen tensions below the critical concentrations, the increase in respiratory quotient indicated the presence of fermentative reactions. The storage life of lemons accompanied by a minimum physiological breakdown was attained at 5 per cent oxygen concentration.

The influence of carbon dioxide on the respiration of citrus fruits in storage has been observed chiefly on lemons. Since this type of information is not available for grapefruit, relevant data are available on the effects of carbon dioxide on the respiration of both climacteric and nonclimacteric fruits (Biale, 1960*a*, *b;* Young *et al.*, 1962). These investigators noted the effects of various concentrations of carbon dioxide at constant oxygen levels on the storage life of lemons, bananas, and avocados; they also determined the respiration rates as functions of carbon dioxide concentration. The respiratory data of the avocado and banana are introduced here to demonstrate the marked differences in chemical behavior between fruits with high respiratory activity and those, such as citrus, with relatively low respiration rates.

In order to compare the effects of various concentrations of carbon dioxide on the respiratory rates of lemon fruits, similar studies have also been reported for the avocado and banana. Since the respiration and ripening of harvested fruits are partially controlled by various gases, it is important to determine what effects carbon dioxide has, when added to the surrounding atmosphere, on prolonging the storage life of fruits at controlled temperatures. This problem was also studied by Young *et al.* (1962), and their data show the effects of carbon dioxide on the relative rates of respiration of the avocado, banana, and lemon. Figure 147 shows the time course of a typical climacteric curve of respiration for the Fuerte avocado under atmospheric conditions (air). This curve serves as a control for the other 2 curves, which illustrate the rates of respiration of avocados in air containing 5 and 10 per cent carbon dioxide. The added carbon dioxide diluted the oxygen concentration in the air; consequently, the required amount of oxygen was added to these gaseous mixtures to return the oxygen level to 21 per cent, the same as in the atmosphere.

When 5 per cent carbon dioxide was added to 21 per cent oxygen, the preclimacteric rate of oxygen uptake by avocados was slightly affected, and the initiation of the climacteric rise was delayed for approximately 3 days. The climacteric peak of respiration was attained after 21 days, a period significantly greater than the 8-day period of the control sample. In the presence

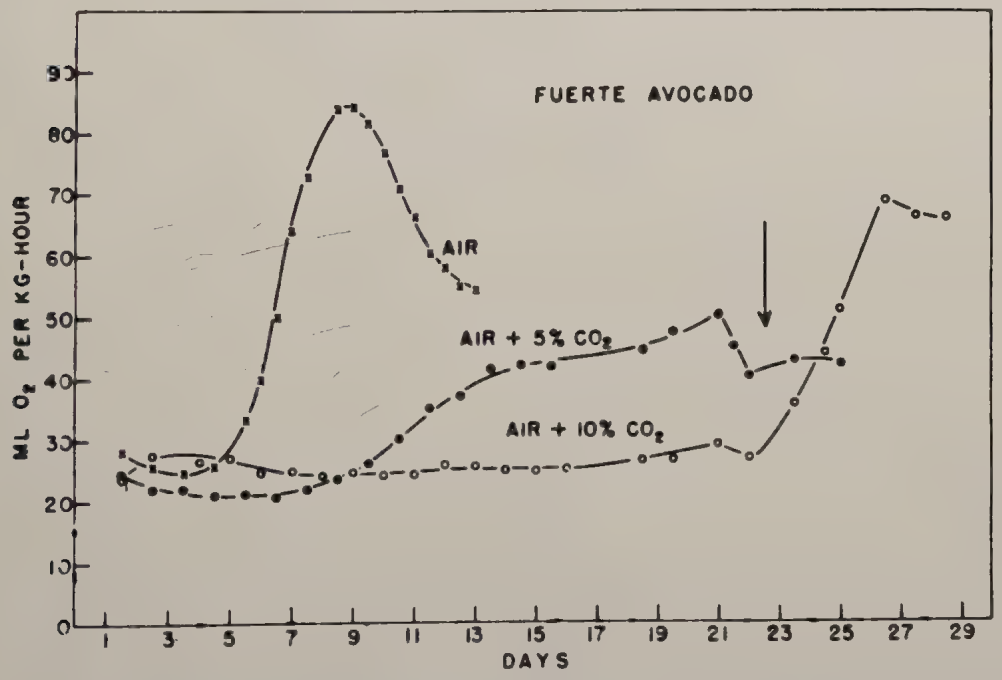


Fig. 147. Oxygen uptake by Fuerte avocado in air and in 21 per cent oxygen with 5 and 10 per cent carbon dioxide at 15° C. (59° F.). The arrow indicates transfer from carbon dioxide treatment to air. (From Young *et al.*, 1962.)

of 5 per cent carbon dioxide, the peak of respiration was 40 per cent lower than that of the air control. In the postclimacteric decline (on the 23rd day), the fruit was transferred to air. The fact that no significant increase in oxygen absorption was observed after the transfer indicated that the fruit had passed the climacteric peak.

On examining the curve in which 10 per cent carbon dioxide and 21 per cent oxygen was used in the storage atmosphere, it can be observed that no change occurred in the respiratory activity of the fruit for 23 days, at which time the rate was equal to the initial value of the control. When the fruit was

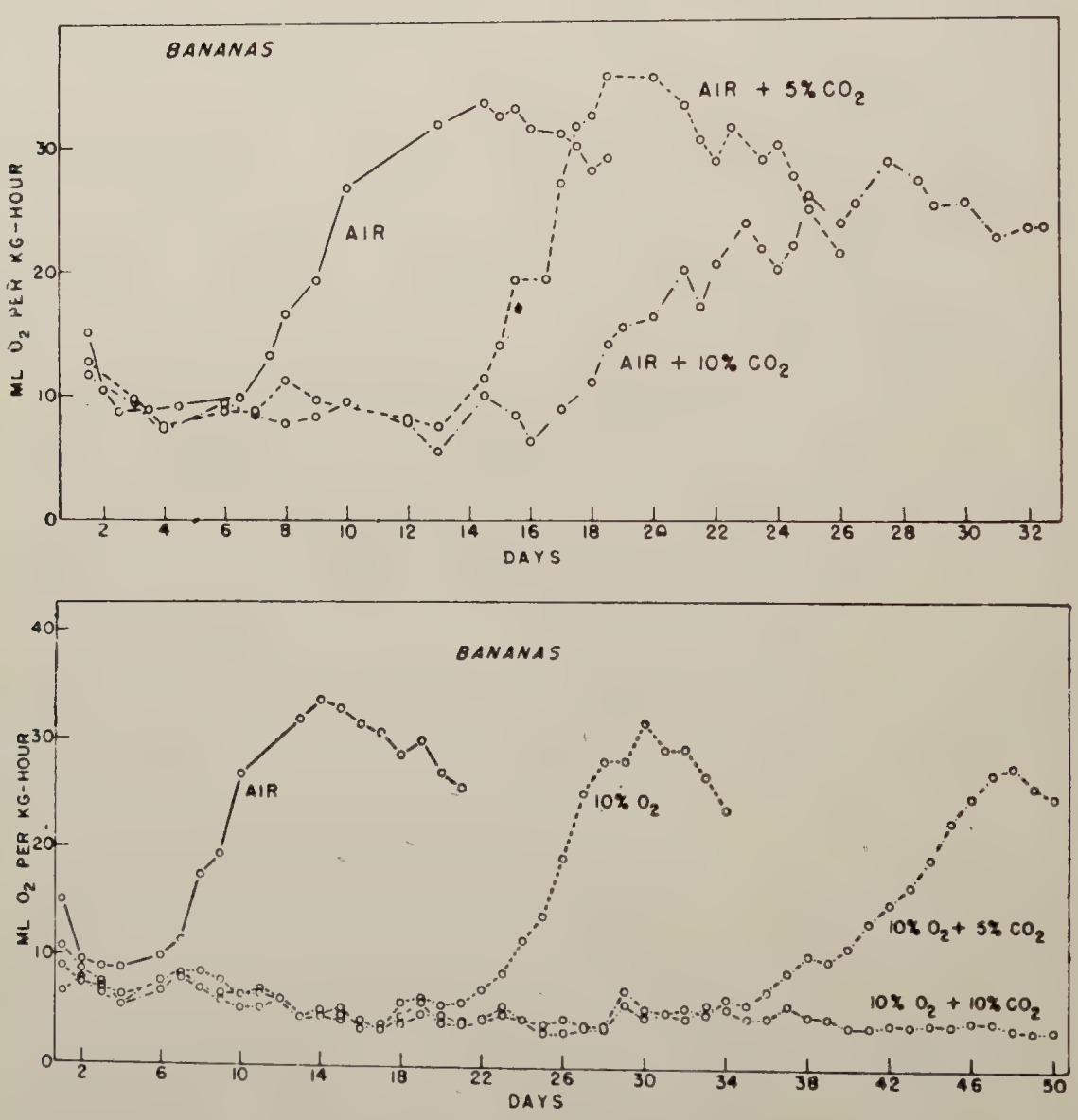


Fig. 148. *Top:* Oxygen uptake by bananas in air and in 21 per cent oxygen with 5 and 10 per cent carbon dioxide at 15° C. (59° F.). *Bottom:* Oxygen uptake by bananas at 15° C. in air, 10 per cent oxygen, and in 10 per cent oxygen with 5 and 10 per cent carbon dioxide. (From Young *et al.*, 1962.)

transferred from the carbon dioxide treatment to air, the rate of respiration increased, indicating the approach of the climacteric peak which was highly repressed under the carbon dioxide treatment (figure 147).

Bananas are analogous to avocados in that they exhibit the climacteric upsurge in carbon dioxide production, or respiration, associated with the ripening process. The curves in figure 148 (top graph) show the rates of respiration of banana fruits stored in air, 21 per cent oxygen with 5 per cent carbon dioxide, and 21 per cent oxygen with 10 per cent carbon dioxide at 15° C. (59° F.). All 3 curves had similar shapes with a definite climacteric peak, and the maximum values of oxygen uptake were of the same order of magnitude. The distinctive differences shown by the 3 curves consisted in the time of initiation of the upsurge and the time of appearance of the climacteric peak. The control curve (in air) reached the peak on the fourteenth day; the other 2 curves, which represent fruit under 21 per cent oxygen with 5 and 10 per cent carbon dioxide, reached their peaks in 19 and 27 days, respectively. The bottom graph in figure 148 shows the oxygen uptake by bananas at 15° C. in air (control curve). The other 3 curves, listed in order, show the respiration rates in 10 per cent oxygen, and in 10 per cent oxygen with 5 and 10 per cent carbon dioxide, respectively. The respiratory curves determined at 10 per cent oxygen tension and at 10 per cent oxygen with 5 per cent carbon dioxide had approximately the same peak values as the control (air). In relation to the time required to reach the climacteric peak, the fruits treated with 10 per cent oxygen required 30 days, those treated with 10 per cent oxygen with 5 per cent carbon dioxide required 47 days, and finally, the fruit in a 10 per cent oxygen and 10 per cent carbon dioxide environment showed no increase in oxygen uptake for 50 days; the fruit remained in a dark-green condition without the appearance of injury. Although avocados and bananas have climacteric patterns of respiration, they respond in different ways to alterations in the gaseous mixtures in which they are stored. In the banana, the initiation of the climacteric was delayed in time by a decrease in the oxygen concentration and by an increase in carbon dioxide content in the storage atmosphere, but these gaseous mixtures had no effect on the rate of respiration. In the avocado, however, the response was different in that the rate of respiration throughout the climacteric was affected by gaseous mixtures of the same concentrations.

Lemons did not give the same response to 5 and 10 per cent concentrations of carbon dioxide in the storage atmosphere as did the climacteric fruits (avocado and banana). The differences are manifested in the curves of figure 149, which show the effects of carbon dioxide on the respiration of lemons at a constant oxygen tension (Young *et al.*, 1962). A pronounced stimulation of oxygen uptake was observed when 5 and 10 per cent carbon dioxide was added to air. For the first 3 weeks, however, the oxygen uptake of all 3 samples

decreased to a minimum. The rate of respiration in air, which is a typical respiratory curve for lemons, continued to decrease slowly for the next 21 weeks.

The curves representing the 5 and 10 per cent carbon dioxide concentrations show the rates of respiration increasing to a maximum at about 9 weeks and decreasing thereafter, but always remaining above the control curve (air). At peak values, the respiratory rates in the 5 and 10 per cent carbon dioxide treatments were respectively 31 and 50 per cent above that of the control curve.

This particular characteristic of lemons—to consume oxygen under relatively high concentrations of carbon dioxide (10 per cent)—led Biale (1960*a*) to investigate the biochemical mechanism that might explain this stimulation. He exposed lemons for 5 minutes to an atmosphere containing radioactive carbon dioxide ($^{14}CO_2$). After the exposure period, the carbon dioxide was rapidly swept from the chamber, the peel was separated from the fruit, and ground in a blender in 70 per cent alcohol. The lipid-free extract was then analyzed chromatographically. The data revealed that 90 per cent of the activity of lemons that were treated with radioactive carbon dioxide for 5 minutes was present in citric, malic, and aspartic acids. This suggested that oxalacetate, which is partially labile and difficult to identify, was the initial compound to receive radioactivity according to the scheme in figure 150.

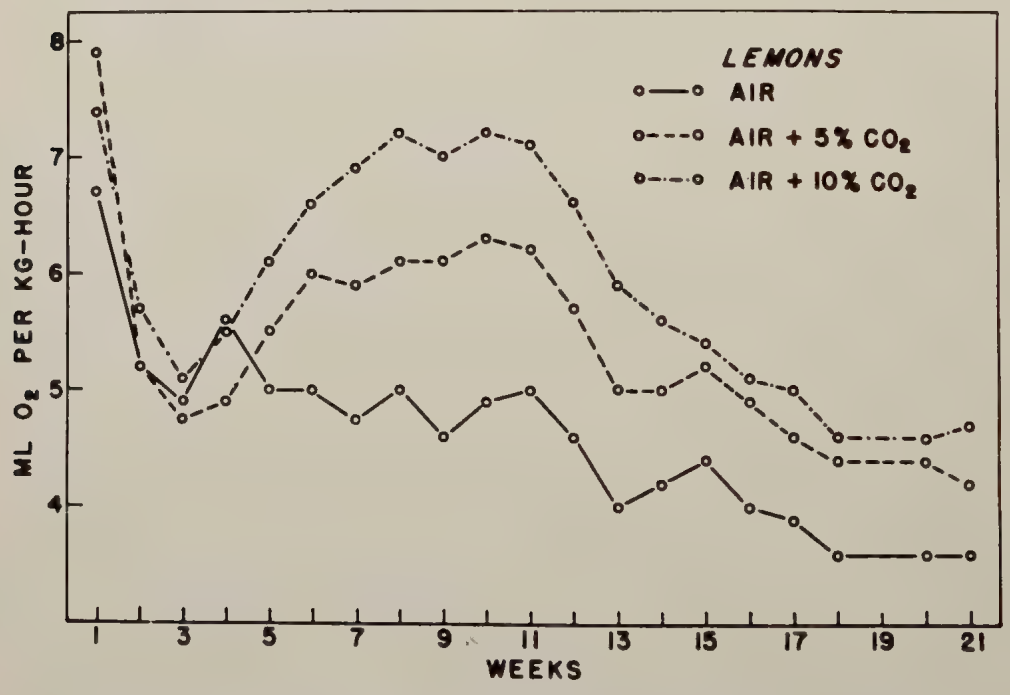


Fig. 149. Oxygen uptake by lemons in air and in 21 per cent oxygen with 5 and 10 per cent carbon dioxide at 20° C. (68° F.). (From Young *et al.*, 1962.)

Biale (1960*a*) also noted that although the enzymes necessary to carry out the reactions in the above scheme had been reported in various organisms, their presence in lemon peel had not been determined, nor had oxalacetate been found to occur in lemon peel, but that this compound would be easily decomposed in the process of isolation.

Allentoff *et al.* (1954) noted that apples could fix carbon dioxide ($^{14}CO_2$), incorporating 64 per cent of the activity as malic acid, and that the fixation increased with an increasing concentration of carbon dioxide in the environment. Definite information is needed on metabolic pathways, which means data on the enzyme systems involved in these reactions, before the mechanisms

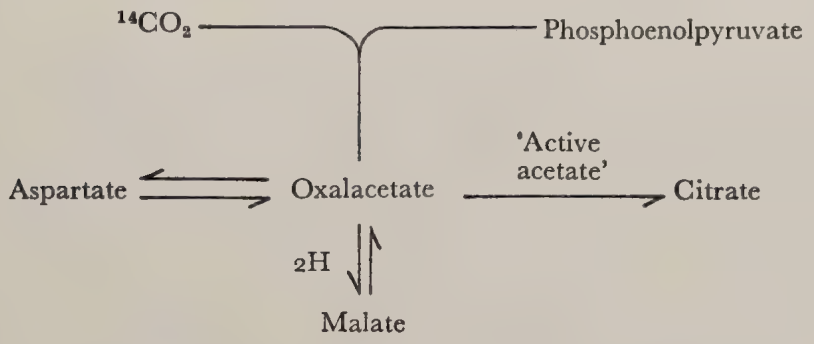


Fig. 150. Suggested scheme for the labeling of organic acids by the fixation of $^{14}CO_2$ through single-step reactions from oxalacetate. (From Biale, 1960*a*.)

of carbon dioxide fixation in the fruit can be explained. When lemons were exposed by Biale to $^{14}CO_2$ for 24 hours, activity appeared in serine, aspartic acid, fats, saponins, and malonic acid. (See also, Young and Biale, 1968.)

In the storage of lemons, Biale and Young (1962) reported that atmospheres containing 5 and 10 per cent carbon dioxide suppressed chlorophyll destruction. For any given concentration of oxygen, the samples in atmospheres low in carbon dioxide showed an accelerated rate of fruit coloring greater than samples stored under higher concentrations.

The cells of fruit absorb gases from the environmental atmosphere through the intercellular spaces which, in some instances, may occupy a considerable portion of the total fruit volume (20 per cent in apples). These intercellular spaces are very important in assisting gaseous diffusion into the tissue cells of the fruit. The internal gaseous composition of the fruit is related not only to the external atmospheric environment, but to the rates and types of metabolic reactions of the fruit cells.

Scholz *et al.* (1960) issued a progress report on the storage of Texas red grapefruit in modified atmospheres. Ruby Red grapefruit was harvested from a commercial orchard early in the season (November 13) and in midseason (February 9). Each sample lot (30 fruits) was subjected to 27 modified atmos-

pheric treatments, consisting of all combinations of 3 temperatures, 3 concentrations of carbon dioxide, and 3 concentrations of oxygen. The atmospheric composition in the storage chamber was changed by displacing the air with nitrogen until the oxygen concentration was reduced, and subsequently introducing the desired amount of carbon dioxide. No decay developed in the early-season fruit stored for 6 weeks, but significant amounts of rot did appear during the 1-week holding period at 70° F. (21° C.), especially in those samples stored at 33° F. (just above zero C.) in high concentrations of carbon dioxide (10.5 to 12.5 per cent). In this particular experiment, the number of decayed fruits varied from 7 to 21 out of a total of 30 fruits per sample.

In the early-season experiments, fruits stored at 50° F. (10° C.) were practically free of rind breakdown, which included pitting, aging, and red blotch. A high degree of pitting occurred in fruits stored at 40° F. (4.4° C.) in air (8 to 10 per cent surface breakdown), and in those atmospheres controlled at high oxygen levels (28 to 55 per cent surface breakdown). Significantly high percentages of surface breakdown were observed in fruit at high carbon dioxide concentrations at both 40 and 33° F. storage temperatures. These rind defects were large, brown, scaldlike areas similar to red blotch, and may have been related to the prevailing wet season. Rind breakdown was severe at all controlled atmospheres at 33° F., except in the experiments at low carbon dioxide levels (2.5 to 4.5 per cent). In early-season fruit, the flavor of the juice was acceptable in all fruit samples stored at 50° F. Off-flavors were detected, however, in fruit stored in high concentrations of carbon dioxide at 40° F. The flavor defects of individual fruits stored at 33° F. appeared to be associated with surface breakdown.

In controlled-atmosphere storage at 50° F., grapefruit, at low oxygen tensions (2.5 to 5.5 per cent), retained the original greenish color; those samples stored at intermediate oxygen concentrations were yellow; and the fruit placed under high oxygen tensions, as well as that stored in air, had the orange-yellow typical of stored fruit. Fruit stored at 33 to 40° F. retained the original greenish cast, while that stored at 40° F. in atmospheres low in oxygen and carbon dioxide tensions maintained its color for several weeks after storage, finally turning yellow, but not orange.

In midseason samples, decay was considerably greater in fruit stored in controlled atmosphere than when stored under normal atmospheric conditions for an 18-week period. The high incidence of decay was probably caused by high humidity in the storage chambers, in which there occurred no circulation of the atmosphere; with small temperature variations, moisture condensed on the fruit surface. In the control experiments, the cartons containing the fruit were surrounded with circulating air at 85 per cent relative humidity. Treatments at high carbon dioxide tensions produced 100 per cent

fruit decay at 40 and 50° F. storage temperatures. Similar treatments at intermediate carbon dioxide concentrations produced 152 decayed fruits out of 180 (84 per cent); and the low carbon dioxide tension experiments resulted in 124 out of 180 (69 per cent). Fruit in cartons in normal atmosphere had 43 out of 120, or 36 per cent decay.

According to Haller *et al.* (1945), it is generally assumed that the rate at which a fruit respires after being harvested is a measure of its deterioration. Respiration is a chemical process, and the rate increases greatly as the temperature rises (figure 151). However, the storage life of the fruit does not

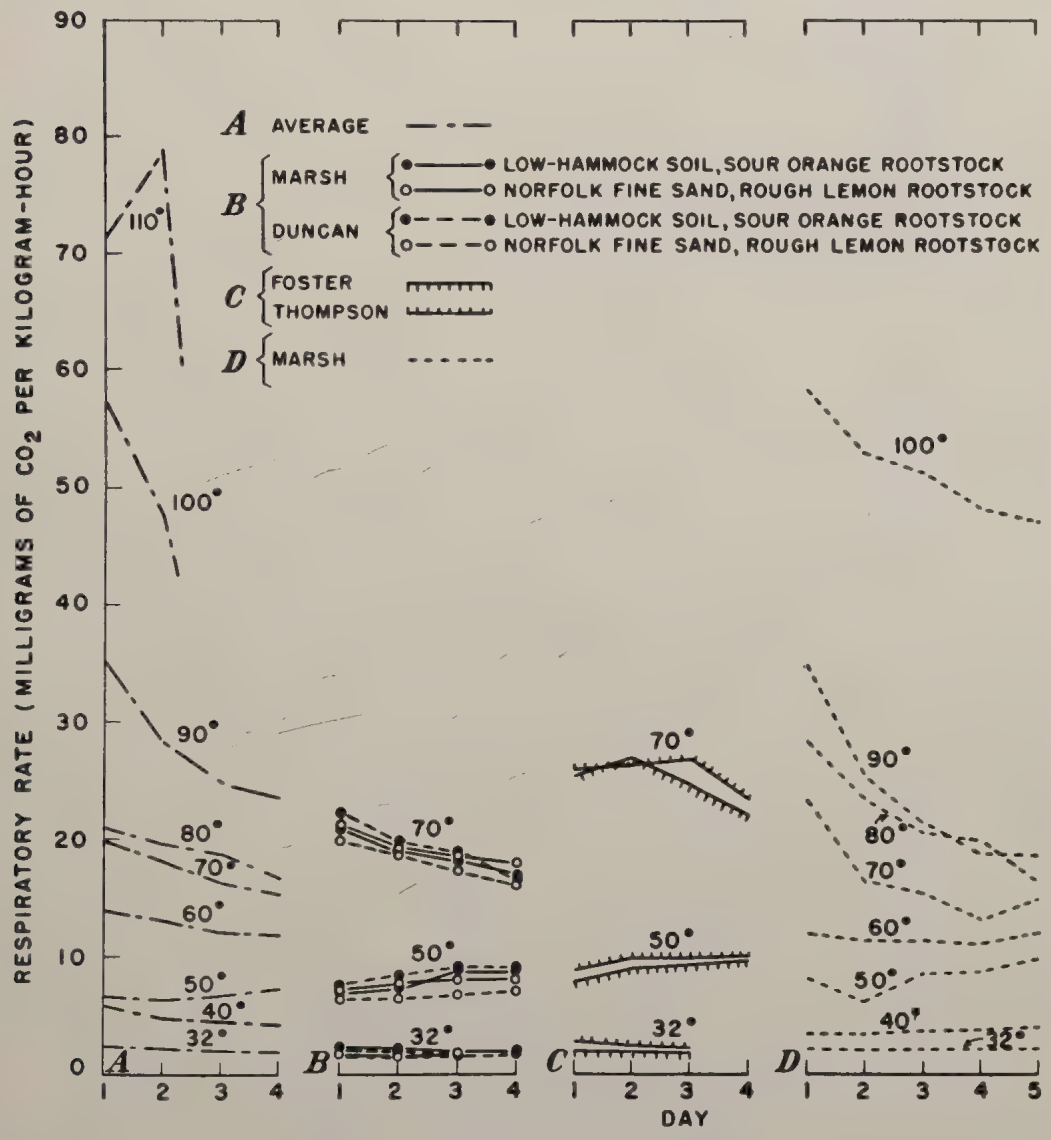


Fig. 151. Effects of temperature (stated as degrees F.) on the respiratory activity of grapefruit. *A*, averages for various lots grown in Florida in the 1929–30 season; *B*, grown in Florida in the 1942 season; *C*, averages for various lots grown at Bradenton, Florida, on sour-orange rootstocks in the 1942 season; *D*, grown in California in the 1932 season. (From Haller *et al.*, 1945.)

necessarily decrease as the temperature rises. For example, certain physiological disorders, such as pitting of grapefruit and other citrus fruits, are associated with relatively low temperatures. Although the rate of respiration of these fruits is greater at higher temperatures, their commercial life is greater at higher than at lower temperatures. On the other hand, the rate of respiration does indicate the rate at which the elaborated foods are being oxidized and the rate at which the fruit is deteriorating in food value. The respiratory rate is a measure of the heat evolved by the oxidation, primarily, of sugars and acids. The amount of heat released is directly important in relation to refrigeration of the fruit in storage and in transit.

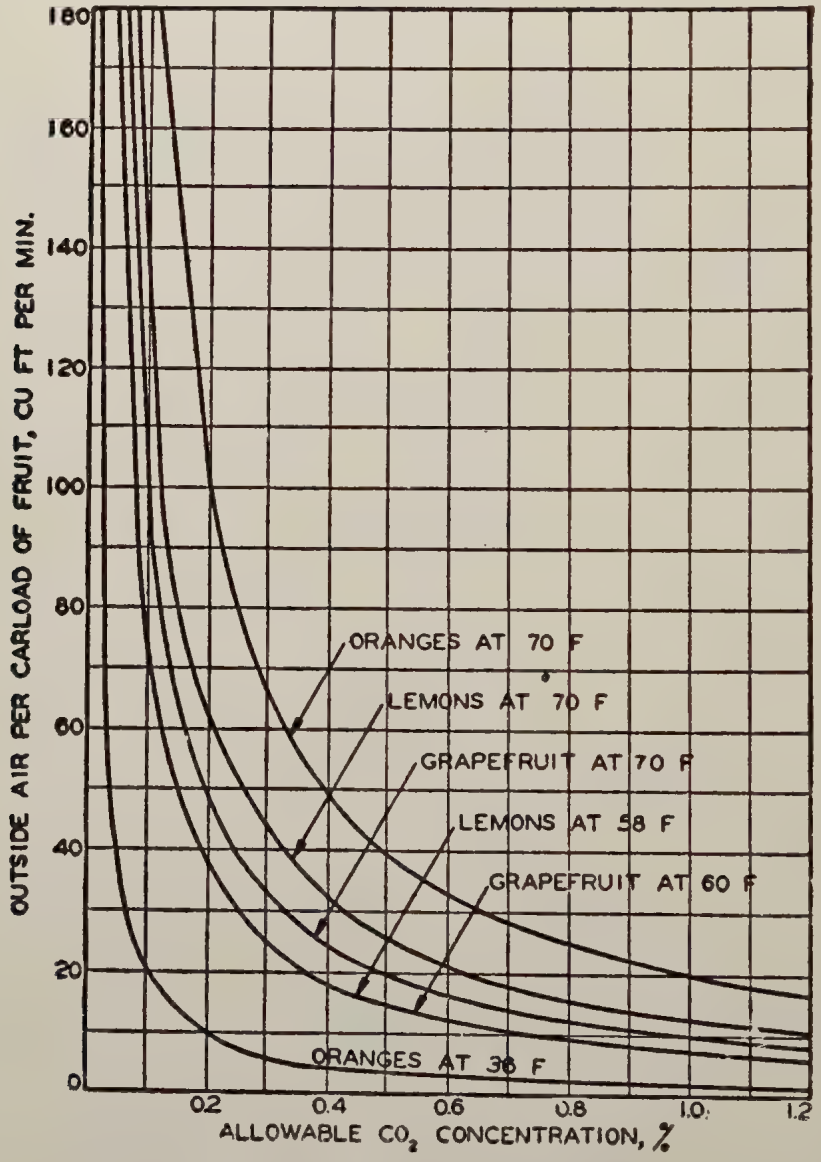


Fig. 152. Outside air requirements for diluting the carbon dioxide in storage-room atmosphere. (From Hendrickson and MacRill, 1948.)

In the storage of grapefruit, Hendrickson and MacRill (1948) stressed the importance of regulating the gases of respiration in the storage room. They concluded that the concentration of carbon dioxide in the storage room would be the best index of other harmful gases of respiration, and that although these gases might exist in relatively small amounts in the storage atmosphere, they would hasten the ripening and impair the keeping qualities of the fruit. To dilute these gases of respiration, the storage rooms would have

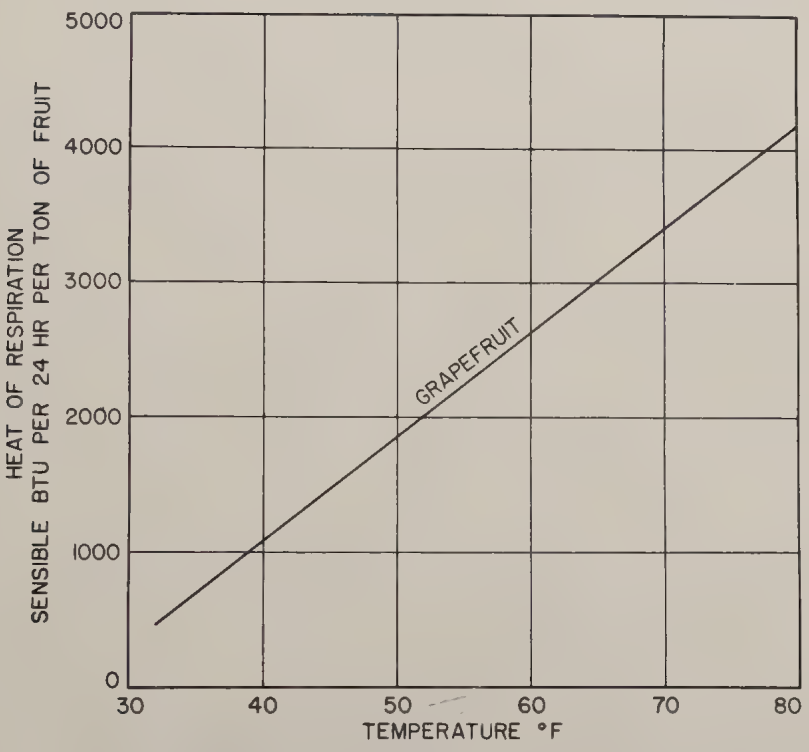


Fig. 153. Heat of grapefruit respiration at different temperatures. (Adapted from Hendrickson and MacRill, 1948.)

to be ventilated with sufficient outside air to maintain the concentration of carbon dioxide at approximately 0.1 per cent. The curves in figure 152 show the amounts of outside air that were required to ventilate citrus storage rooms in order to maintain the desired carbon dioxide concentration. For grapefruit at 60° F. and for lemons at 58° F. (16 and 14.4° C.), the outside air requirements were 37 cubic feet per minute per carload for 0.2 per cent and 72 cubic feet per minute for 0.1 per cent carbon dioxide.[3]

Hendrickson and MacRill (1948) also constructed a graph showing the heat of respiration of grapefruit in relation to the temperature (figure 153). During the respiration process, the oxygen of the air reacted with the carbon of the sugars to form various decomposition products, and finally carbon

[3] One cubic foot equals 28.3 cubic decimeters.

dioxide and water. During the oxidation of the sugars, energy was found to be released in the form of heat, and the amount increased with the rise in temperature up to about 100° F. (37.8° C.). In general, grapefruit showed a lower respiration rate than lemons and oranges (Valencias and Washington Navels). The data in table 173 show the heat of respiration (sensible heat) that was calculated from the rate of production of carbon dioxide from grapefruit in storage. Values are also reported for the quantities of sugar oxidized for the equivalent amounts of carbon dioxide produced.

Harvey and Rygg (1936*b*), in California, devised a special apparatus by means of which they could determine the vitality of fruit. They placed the fruit in an airtight chamber, and determined the negative or positive pressure produced by respiration. A great variety of pressure changes (negative or positive) were produced by different individual fruits. Most tests were made at 72° F. (22.2° C.), but some were made at 56, 46, 42, and 38° F. (13.3, 7.8, 5,5, and 3.3° C.).

For all practical purposes, the heat of respiration at a given temperature may be calculated when the respiration rate (milligrams of carbon dioxide per kilogram of commodity per hour) is known. Specific heat is made up of approximately 90 per cent sensible heat and 10 per cent latent heat.

Ethylene

An important role is played in the citrus industry by ethylene, in that it accelerates the coloring of citrus fruits in storage. It is a product of certain storage molds, and when it accumulates in storage rooms at relatively high concentrations, it accelerates the deterioration of the fruit. According to the data of Biale *et al.* (1954) and Pratt (1954), ethylene is a normal metabolic product of many plant tissues, and the amounts present in many fruits have a physiological significance in hastening the ripening process.

TABLE 173

Respiration Data for Grapefruit.*

(All factors are per 24 hours per 2,000 pounds of fruit.)

Storage temperatures (F. and C.)	Sensible heat produced (btu)†	Latent heat produced (btu)	Sugar consumed (pounds)	Moisture produced (pounds)	CO_2 produced (pounds)	CO_2 produced (cu. ft.)‡	Oxygen consumed (cu. ft.)
32 (zero)..	460	32	0.050	0.030	0.074	0.602	0.502
40 (4.4)...	1,070	74	.117	.070	.172	1.423	1.186
60 (16.0)..	2,770	193	.303	.182	.445	3.833	3.194
80 (27.0)..	4,180	291	.458	.274	.672	6.010	5.010

*From Hendrickson and MacRill (1948).
†From Rose *et al.* (1949).
‡Corrected to storage temperatures and atmospheric pressure (29.98 inches of mercury).
Metric conversions: One pound equals 0.454 kilogram; one cubic foot equals about 28.3 dm^3; 29.98 inches equal 76 centimeters.)

For practical purposes, Biale (1960*a*, *b*) divided edible fruits into 2 classes, based on their distinct patterns of respiratory activity. The climacteric type of fruit was stated to consist of those fruits which, on ripening, were associated with a rapid increase in respiration, commonly called the "climacteric rise," and the nonclimacteric type was defined as consisting of those fruits which, on ripening, were associated with a gradual decline in respiration during maturation and senescence. Citrus fruits were regarded as typical examples of the nonclimacteric class, while avocados and bananas would be typical examples of the climacteric category. Biale's data showed that these 2 classes

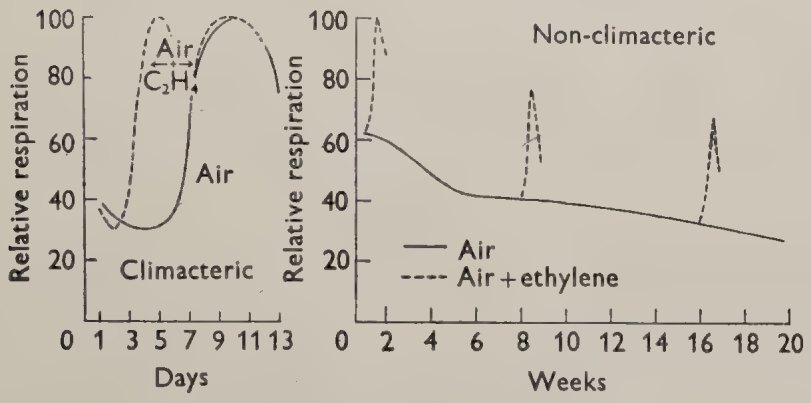


Fig. 154. The responses of climacteric and nonclimacteric fruits to ethylene. (From Biale and Young, 1962.)

responded differently to ethylene. In the climacteric fruit, ripening and respiration were not affected if the fruit was exposed to ethylene after the onset of the "climacteric rise," but ethylene, at 0.1 to 1,000 ppm in air, was effective if applied during the preclimacteric phase.

Biale and Young (1962) stated that ethylene's chief function was to initiate the climacteric rise of respiration by shifting the time axis without effecting any change in the shape of the respiratory curve, and without effecting changes in the major constituents that would not have occurred in the normal course of the ripening process. In nonclimacteric fruits, ethylene was found to accelerate respiration at any time during the life of the detached fruit (figure 154). In citrus fruits, the rate of respiration appeared to be a function of ethylene concentration, at least up to 100 ppm.

Biale (1940) and Biale and Shepherd (1941) reported that a single lemon inoculated with green mold caused rapid coloring and deterioration of about 500 healthy lemons. Later, Biale (1954) extended these studies to Marsh grapefruit. He showed that if a lemon fruit was inoculated with green mold (*Penicillium digitatum*), the emanations or vapors produced caused a marked increase in the respiration rate with deterioration in the keeping quality of grapefruit (figure 155). In these experiments, the moldy lemon fruit was

kept at all times in a separate container from that of the grapefruit samples, and the spore-free air that contained the emanations from the moldy lemon was passed over the sample. Biale concluded that emanations from moldy lemons had an effect on grapefruit and oranges similar to that on lemons. He also noted that green mold was particularly effective in producing ethylene.

Color Development and Degreening of Citrus Fruits. As indicated by research on the germination and growth of grapefruit seedlings, the tempera-

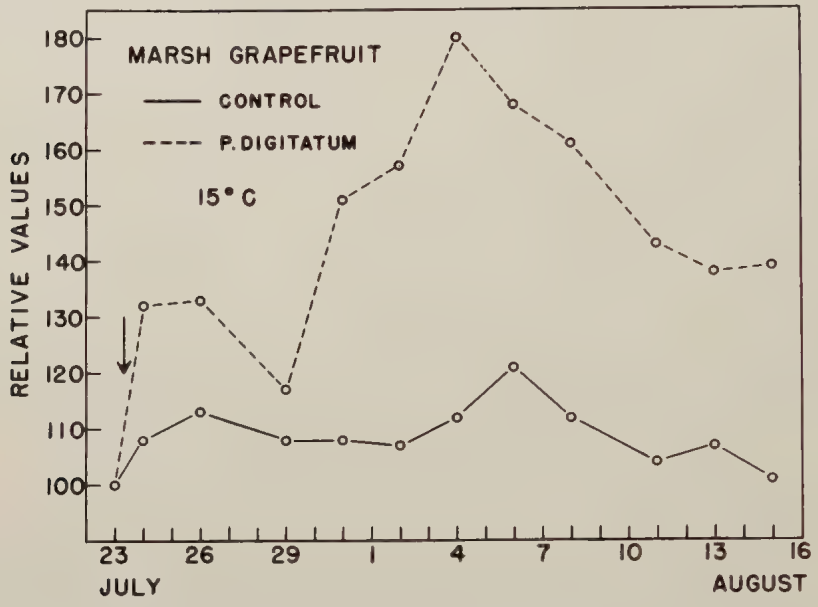


Fig. 155. Effects of emanations from green mold (*Penicillium digitatum*) on the respiration of sound Marsh grapefruit at 15° C. (59° F.). The arrow indicates when mold vapors started. (From Biale, 1954.)

ture at which various metabolic processes become activated and growth begins is approximately 55° F. (13° C.)—the vital temperature of growth. It has been shown experimentally that below this temperature, active growth is rapidly reduced, and dormant plants remain dormant. However, it should be emphasized that at this temperature, or slightly higher, it is possible for some growth activity to appear, for it must be recognized that no single temperature would apply absolutely to all different varieties and species. It can therefore be concluded that climatic and seasonal differences will have marked effects on the color development and degreening of citrus fruits. This statement is especially true with reference to the growth and maturity of citrus fruits as influenced by temperature changes.

During seasonal changes in various varieties of oranges and grapefruit, Stearns and Young (1942) determined experimentally those climatic factors that effected color changes in the fruit. They observed no definite color change in oranges during the fall season until minimum night temperatures

fell below 55° F. (13° C.); the rate of change from green to orange was related to the rapidity of temperature reduction and the continued recurrences of minimum temperatures below 55°. For example, during 1940 the maximum change in color occurred 30 days after the initial break in color, and during this interval, the minimum temperature fell consistently below 55° F. In a similar experiment in 1941, the time required after the first color break to reach the maximum color of the preceding year was 56 days, but during this period the minimum temperature did not fall below 40° F. (4.4° C.).

It is worth noting that the above data were determined solely on oranges (Hamlin, Parson Brown, and Pineapple), and would not be applicable to seasonal color changes in Marsh and Duncan grapefruit.

In the growing seasons of both 1940 and 1941, the color developed very gradually in Marsh and Duncan during the fall and winter months, and at no time during the sampling period was there any visible color break in the fruit, regardless of the minimum temperature. The inference can thus be made that minimum temperatures below 55° F. did not produce an accelerating effect on the color development of these 2 varieties of grapefruit. Color changes in grapefruit would not be expected to be as great as in oranges because grapefruit has smaller amounts of carotenoid pigments, but the chlorophyll showed a gradual disappearance during the sampling period.

Erickson (1960), in studying the effects of temperature on the color development of citrus fruits, reported that Valencia oranges grafted onto small plants developed a uniformly orange color during 2 months' exposure to temperatures of 20° C. (68° F.) days and 5° C. (41° F.) nights. These oranges contained more total pigments and retained less chlorophyll than fruits that did not color appreciably as a result of exposure to either 30° C. (86° F.) days and 10° C. (50° F.) nights, or to greenhouse temperatures with a minimum of 19° C. (66° F.). These results indicated that a high daytime temperature might interfere with fruit coloring. As a result of warmer temperatures, vegetative growth was most abundant on the greenhouse plants.

In another paper, Young and Erickson (1961) extended these studies on grafted fruit to include not only day and night air temperatures, but also soil temperatures. They used Valencia oranges grafted onto Cleopatra mandarin seedlings, on which orange-colored fruits were produced in growth chambers at 20° C. (68° F.) for daytime air, 7° C. (44.6° F.) for nighttime air, and 12° C. (54° F.) for soil. An increase in temperature for any 1 of these 3 variables resulted in fruit with less orange color.

In Arizona, Martin (1942) observed that the rate of color break in Marsh grapefruit was accelerated by a low concentration of nitrogen in the tissues of the tree, irrespective of whether this characteristic was caused by reduced fertilization, time of application of nitrogen, or competition of nitrogen by cover crops.

Grierson and Newhall (1960) noted that evidence was accumulating to support the belief that the kind of fertilizer and the time of its application would indeed affect the degreening of citrus fruits, as well as the color break of the fruit on the tree. Using Valencia orange samples from the Indian River fertilizer plots of Reitz (1957), these researchers found that as the nitrogen application was increased, the percentages of oranges meeting the color requirements for the number 1 grade decreased. On degreening these samples with ethylene, the pack-out was usually increased, except in those fruit samples from the high-nitrogen plots, which apparently were not greatly affected by the degreening process. Furthermore, the results of the studies indicated that the total amount of mixed fertilizers applied to the trees not only affected fruit color, but also affected the subsequent degreening.

Grierson and Newhall (1960) also determined the methods and furnished the necessary information needed to degreen Florida citrus fruits. In many citrus-growing areas, the external color of the fruit is, in some instances, not highly associated with its internal quality. In the absence of cool nights, early-season fruit in Florida may have good eating quality, but may also have an intense external green color. Since the green color of oranges and grapefruit is usually associated with immaturity, it follows that the green chlorophyll can be removed from the peel with ethylene gas without producing any noticeable ripening reactions on the internal fruit tissues, thus making the fruit more acceptable to the consumer. It is our present purpose to report that portion of Grierson and Newhall's data on ethylene degreening which has a bearing on the physiology of grapefruit.

It is well known that stem-end rot is increased when citrus fruits are degreened with ethylene. Without citing *all* the voluminous literature, it can be stated with a considerable degree of confidence that the stem-end rot caused by *Diplodia* is stimulated by ethylene, but the stem-end rot caused by *Phomopsis* is *not* stimulated by ethylene treatments.

Winston and Meckstroth (1944) reported that *Phomopsis* was responsible for most of the rot in untreated lemons, but that *Diplodia* was responsible for the rapid increase in stem-end rot of lemons receiving ethylene treatments. Brooks (1942, 1944) stated that ethylene-treated oranges had 9 times as much stem-end rot as untreated fruit at 2 weeks after harvest, and that this significant increase in rot was caused chiefly by the *Diplodia* fungus. Voorhees (1944) also observed that the increased amounts of stem-end rot in ethylene-treated fruit were caused by the stimulated activity of *Diplodia*. Hopkins *et al.* (1944), basing their conclusions on the results of a large number of experiments, reported that ethylene treatment of oranges increased stem-end rot from a mean value of 20.7 to 47 per cent after 3 weeks from harvest.

Similar results were obtained by Grierson and Newhall (1960), who determined the effects of ethylene degreening on stem-end rot of 10 types of citrus,

TABLE 174

EFFECTS OF DEGREENING ON STEM-END ROT OF VARIOUS CITRUS TYPES, SEPTEMBER 1952 TO MARCH 1955.*
(All samples were held at 70° F. [21° C.] until 2 weeks from picking date.)

Types of fruit	Number of experiments	Total number of fruits	Average losses from stem-end rot (per cent)		Increases in stem-end rot owing to degreening (per cent)	Analyses of variance: *F* values
			Not degreened	Degreened		
Tangerine	27	8,100	12.8	26.1	204	60.87*
Hamlin orange	18	2,700	5.1	17.8	350	8.97*
Valencia orange	17	2,250	7.1	17.4	245	22.32*
Parson Brown orange	2	300	26.0	35.5	135	....**
Pineapple orange	1	150	8.0	60.0	750	...**
King "orange"	1	150	29.0	60.0	206	...**
Temple orange	14	2,100	11.3	31.2	276	7.48†‡
Duncan grapefruit	18	1,080	3.2	10.7	335	53.86*
Marsh Seedless grapefruit	2	120	0.0	8.5	...	...**
Foster Pink grapefruit	2	120	1.0	9.5	950	...**
Totals	102	17,070				
Weighted averages			8.51	21.3	251	

*From Grierson and Newhall (1960).

Key to terms in the last column: One asterisk means "significant at the 1 per cent level"; two asterisks mean "too few experiments for statistical evaluation"; a dagger means "significant at the 5 per cent level" (see Grierson and Newhall's table 1); a double-dagger means "losses from stem-end rot reduced (hence statistical significance reduced) owing to losses from peel injury before the second-week examination." (See also, Grierson and Newhall's figure 23.)

including grapefruit, for 3 seasons. Their data showed that degreening with ethylene increased stem-end rot about 2.5 times more than that in the control samples when held for 2 weeks after harvesting at 70° F. (21° C.) (table 174). The rate of acceleration of stem-end rot, as shown in the table, was the result of all the variables involved in the degreening process, since the control samples were exposed to all the degreening room conditions except ethylene.

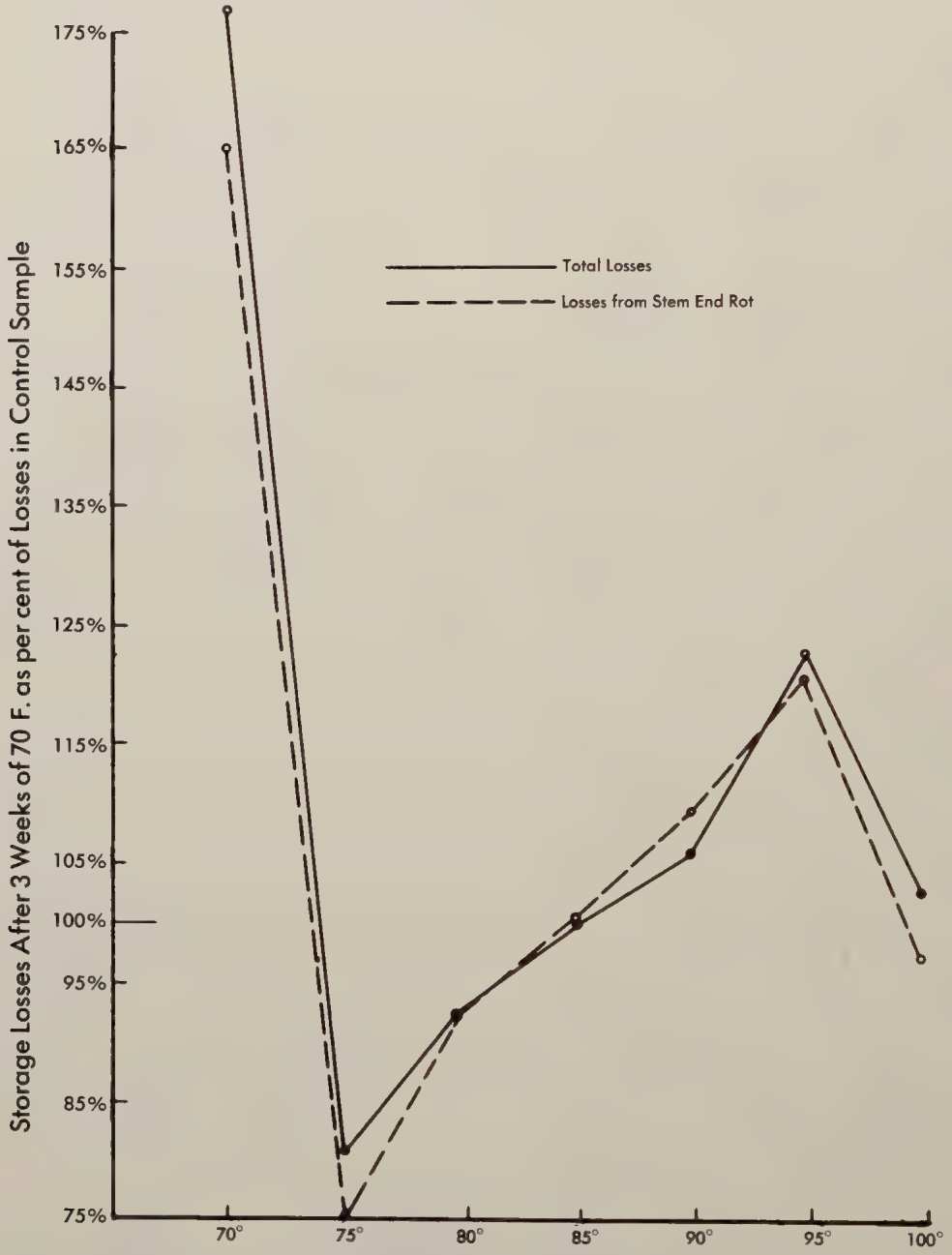


Fig. 156. Storage losses from fungi after 3 weeks at 70° F. (21° C.) in Marsh grapefruit degreened at various temperatures, shown as percentages of losses recorded in control sample. (From Grierson and Newhall, 1953.) Temperatures are given in degrees Fahrenheit.

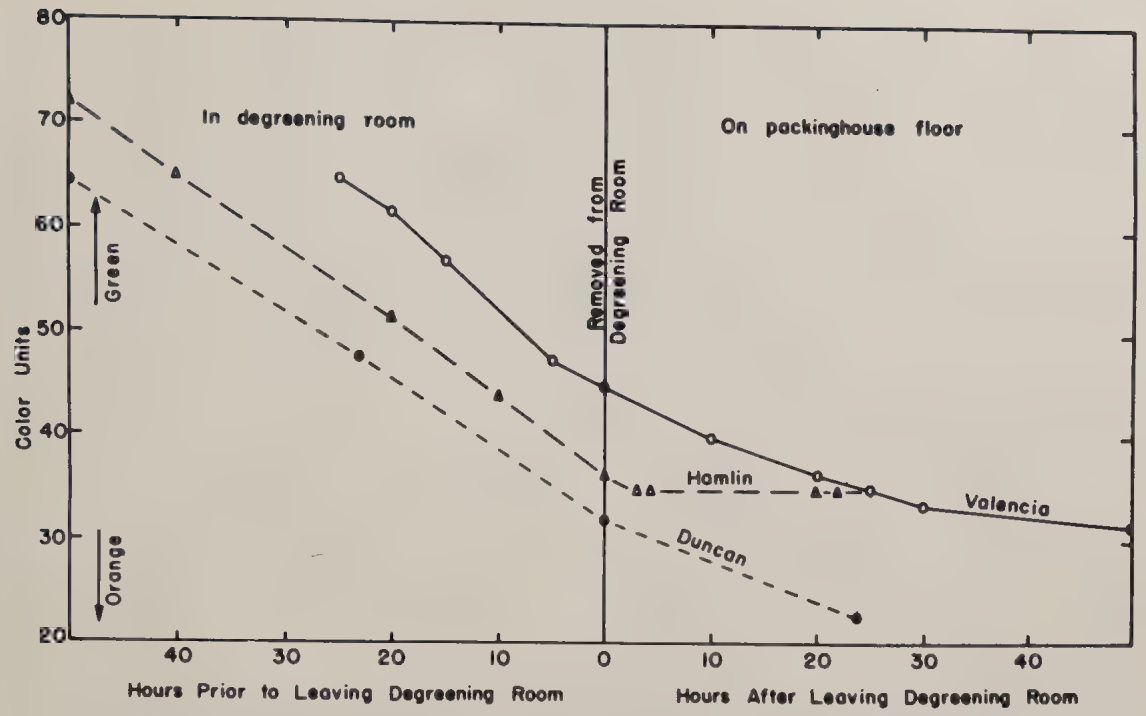


Fig. 157. Rates of degreening of Florida oranges and grapefruit before and after removal from degreening room. (From Grierson and Newhall, 1960.)

Grierson and Newhall (1953), after degreening grapefruit with ethylene, stored samples at 70° F. (21° C.) for 3 weeks for the purpose of observing, at stated intervals, the amounts of decay from stem-end rot and other fungi. An inspection of the curves in figure 156 will show the association between degreening temperatures and the percentages of fungal decay in grapefruit. Since susceptibility to fungi varied with fruit maturity, they reported losses as percentages of the control samples degreened at 85° F. (29.4° C). The 2 curves, representing total loss and loss from stem-end rot, are hardly distinguishable, which strongly indicates that the fungal loss of fruit in storage was caused chiefly by stem-end rot, with a small percentage of rot caused by other fungi, principally *Penicillium*. It can be observed that any increase in degreening temperature above 85° F. caused a corresponding increase in fungal decay of grapefruit as well as excessive shrinkage.

It should be understood that degreening of the fruit may occur on the tree, in cool or cold storage, or during treatment with ethylene. Ethylene merely accelerates changes that would occur naturally later, whether or not it is used. Oberbacher *et al.* (1962) reported that early-harvest, nonethylened, nonwaxed Marsh Seedless grapefruit shipped to European ports in ventilated holds would successfully degreen en route, and with less pitting and decay than ethylene-treated fruit. By eliminating the standard ethylene degreening procedure at 85° F., the amount of decay which frequently occurs in long overseas shipments was significantly reduced.

In order to prevent pitting, grapefruit is often permitted to stand for a "hardening" period after degreening before the fruit is processed in the packing house. Some varieties of citrus fruits continue to change in color after removal from the presence of ethylene. In the Florida experiments of Grierson and Newhall (1960), Duncan grapefruit continued to change color at approximately the same rate for 24 hours after removal from the degreening rooms, while color development in the Hamlin orange was discontinued within 6 hours after completion of the ethylene treatments. After a similar degreening process, Valencia oranges continued to develop color for about 30 hours (figure 157).

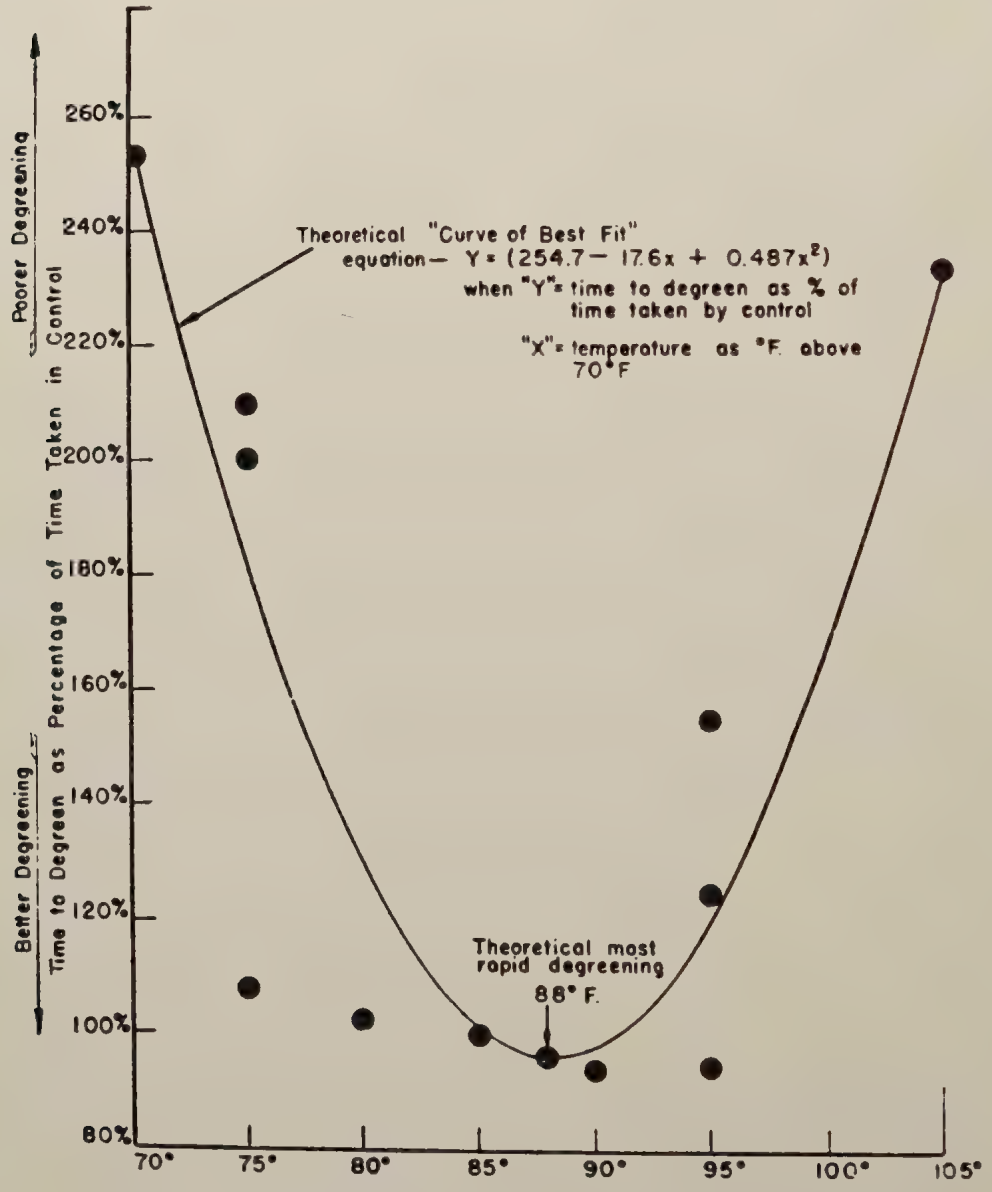


Fig. 158. Effects of temperature on the rates of degreening of Marsh grapefruit. (From Grierson and Newhall, 1953.)

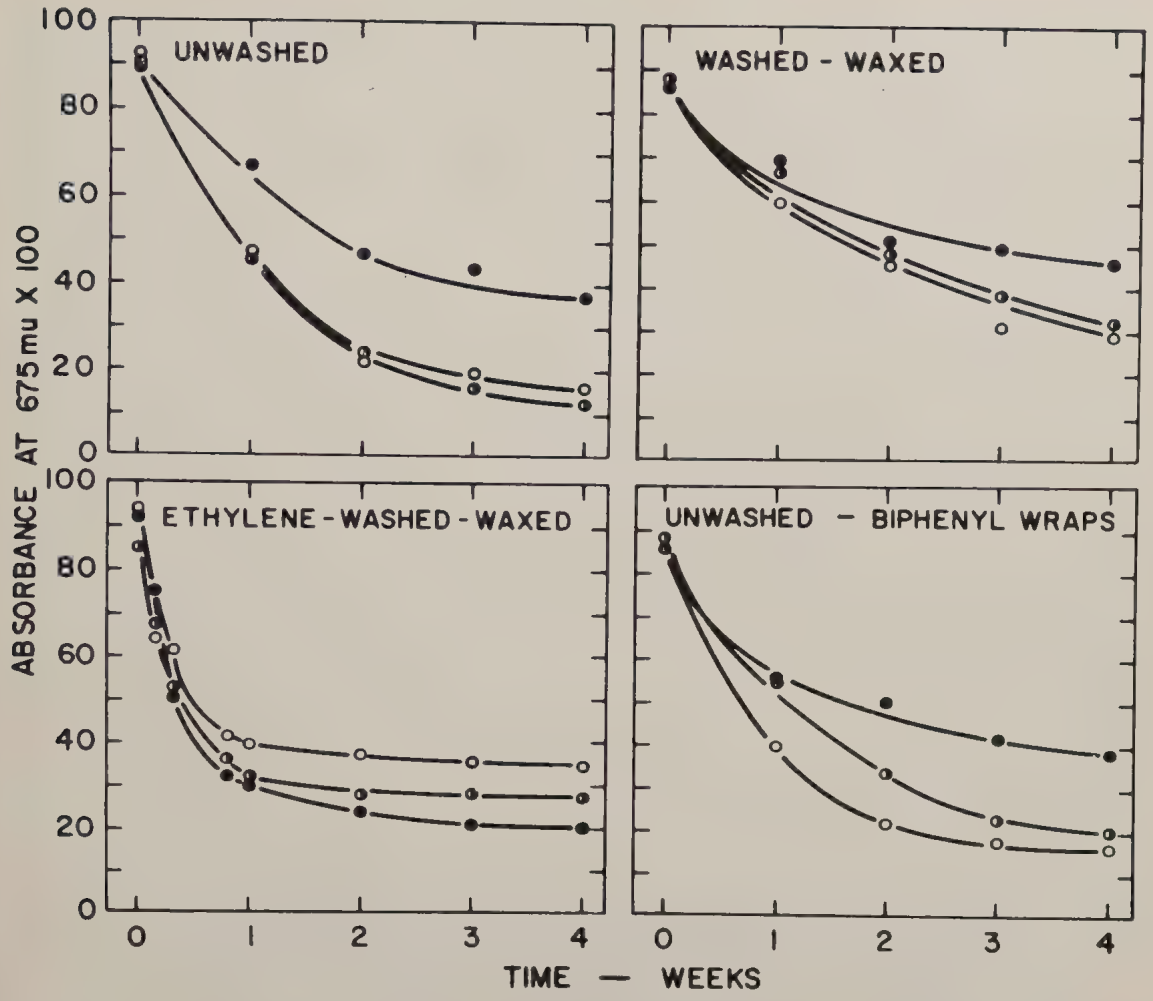


Fig. 159. Degreening of dark-green Ruby Red grapefruit peel from harvest through degreening, handling, and storage times. The ethylene-treated fruits were degreened at 85° F. (29.4° C.) for the first 5 days before being packed and held at the indicated storage temperature. Black circles indicate 50° F. (10° C.); open circles, 60° F. (16° C.); half-black circles, 70° F. (21° C.). (From Oberbacher, 1962.)

Grierson and Newhall (1953), in studying the effects of temperature on the rates of degreening, noted that the experimental data obtained on Marsh grapefruit were much less consistent than those for oranges. The curve in figure 158 clearly illustrates that an optimum temperature was present, which indicates that the rate of degreening was reduced at temperatures above or below this maximum value. Owing to the relatively wide range in temperature near the optimum value, it was thought best to plot a theoretical "curve of best fit" rather than to use the curve formed by joining the experimental points. The most rapid degreening temperature for Marsh grapefruit occurred theoretically at 88° F. (31° C.)—a little higher than the 85° F. (29.4° C.) that proved to be the optimum temperature for both Valencia and Hamlin oranges.

Oberbacher (1962) reported on the rate of color change of the peel of Ruby Red grapefruit as influenced by temperatures and handling practices (figure 159). In this time-course study, the initial fruit samples had highly green-colored peels, as indicated by the high absorbance values at zero time. Treatment with ethylene for 5 days at 85° F. did not entirely eliminate the green color, but the fruit did show a rapid change in peel color, as indicated by the abrupt decline in the absorbance values for the first week in storage at 50, 60, and 70° F. (10, 16, and 21° C.). The absorbance values of the unwashed fruit, with or without biphenyl-treated wraps, declined at a slower rate than those of the ethylene-treated samples, but the color of the unwashed fruit was equal

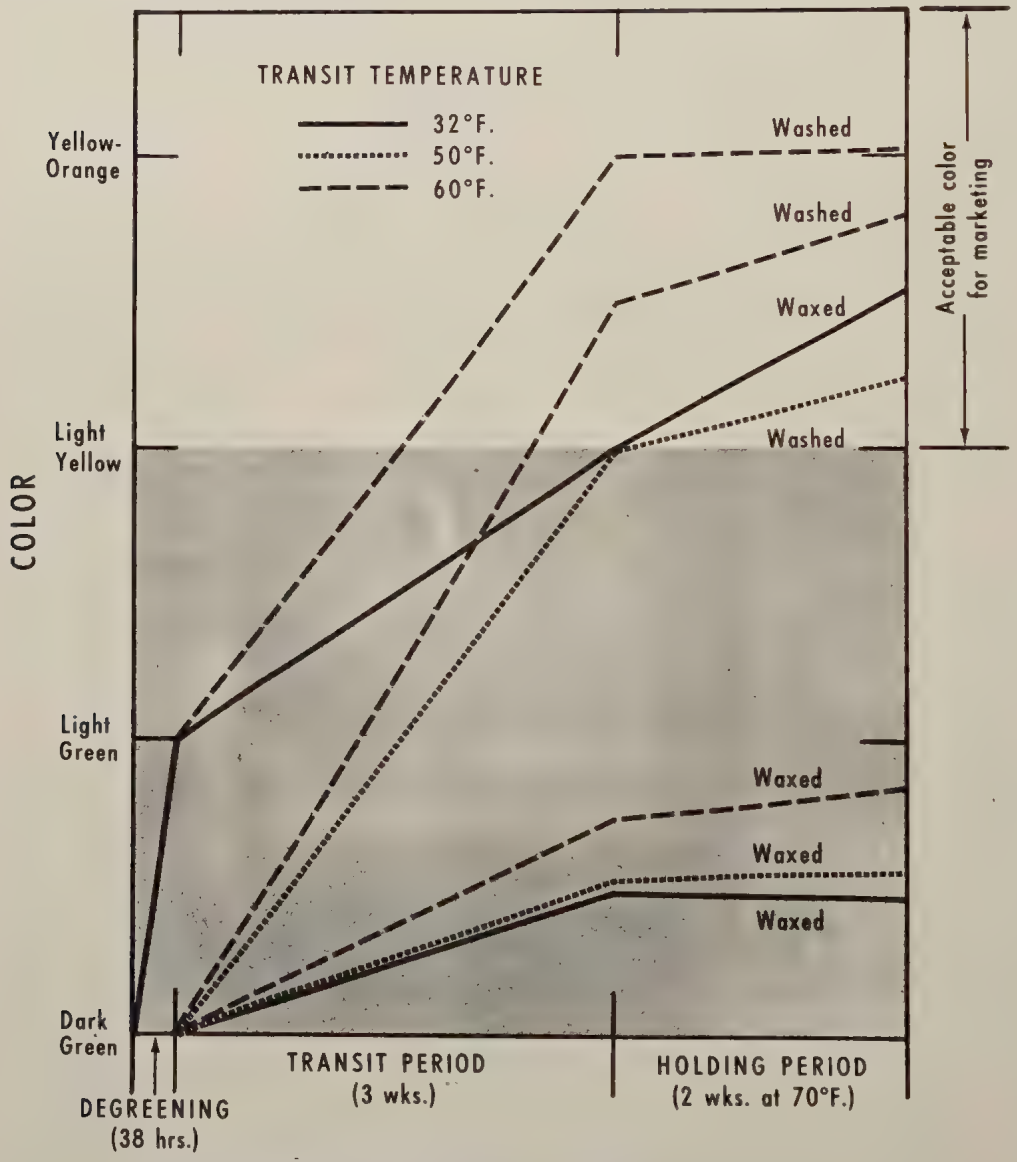


Fig. 160. Effects of pretransit treatment and export transit temperatures on the degreening of Marsh grapefruit. (From Chace *et al.*, 1966.)

to that of the treated fruit after 2 weeks of storage, and at the end of 3 weeks, the fruit stored at 60 and 70° had developed an acceptable color. Waxed fruit degreened at a comparatively slow rate at all storage temperatures, as demonstrated by the fact that it retained about 40 per cent of its green color after 4 weeks in storage. In these experiments, the storage temperature of 50° reduced the rate of degreening. It should be noted that the degreening rate is also a function of the initial intensity of the green color in the sample.

Chace *et al.* (1966) investigated the effects of pretransit treatments and export transit temperatures, as well as holding periods, on the rates of degreening of early-season Florida grapefruit. The degreening of postharvest grapefruit by some adequate procedure is essential from September to January if it is to be successfully marketed in the fresh state. Consequently, the biochemical changes that grapefruit undergoes during the time intervals between harvest and arrival in the foreign markets are highly related to the quality of the fruit. In terms of color development under postharvest conditions, the data for Marsh grapefruit are illustrated in figure 160. Dark-green, waxed grapefruit, without ethylene treatment, did not degreen sufficiently to attain a light-green color during the 3-week transit period at 32, 50, and 60° F., respectively, and the 2-week holding period at 70° F. (zero, 10, 16, and 21° C.). Washed fruit, without waxing or degreening, developed a yellow to yellow-orange rind color, the acceptable color for marketing, during the 3-week transit period at 50 and 60° F. Samples that received the 36- to 38-hour ethylene treatments changed from dark to light green (see the figure). After degreening, the peel of both the washed and the washed and waxed fruit changed from light yellow to yellow-orange during the combined 5-week transit and holding periods. December-harvested grapefruit that progressed naturally on the trees to a light-green color required 38 hours of ethylene treatment to develop a light-yellow peel. Fruit degreened with ethylene before transit had a light-yellow rind color which developed to yellow-orange after the transit and holding periods.

Winston (1942) reported that oil sprays prevented the development of color while the fruit was on the trees as well as in the degreening rooms.

Biphenyl as a Fungistat

GREEN-MOLD ROT of oranges was found to be efficiently controlled by biphenyl, when used as an impregnating material in tissue wraps, as reported by Dr. R. G. Tompkins (1935), of the Low-Temperature Research Station, Cambridge, England. He noted that biphenyl-treated wraps not only reduced the number of rotting fruits, but also that spore formation usually accompanied rotting.

Adalbert Farkas (1938), of the Hebrew University, Palestine, reported that biphenyl-impregnated wraps on Palestine-grown oranges gave remarkable green-mold control. Oranges in nontreated wraps showed 6 to 7 times more

decay than those in biphenyl-treated wraps. Also, he claimed that no taint in flavor was caused by the wraps, and that the odor disappeared after 2 or 3 weeks in storage.

Dr. Tompkins, during a visit to southern California, furnished some interesting information about the early use of biphenyl as a preservative. Soon after the extremely encouraging results reported by Adalbert Farkas, shipments of oranges in biphenyl wraps began arriving in England from Palestine. The pungent odor emitted by the wraps came to the attention of British health officers at ports of entry, and without investigation of the possible harmfulness of biphenyl to humans, but with plenty of suspicion and armed with the phrase "medicated fruit," they forbade further entries of such shipments. At this juncture, Dr. Tompkins found himself in an uncertain situation regarding this aromatic compound, and all efforts to remove the injunction failed. However, Mr. Farkas, in response, went to England, and succeeded first in acquiring the biphenyl patent rights from Dr. Tompkins, and then proceeded to continue the shipments of oranges in treated wraps—but still without the injunction having been removed! Although the edict continued in effect for a considerable period, it appears that fruit from all the major citrus-growing areas of the world continued unofficially to arrive in England unhindered.

In 1938, the Field Department Laboratory of the Sunkist Growers, with Crown Zellerbach in cooperation, participated in experiments to determine the value of biphenyl as a mold control. This marked the beginning, in the United States, of innumerable tests concerned with the action of biphenyl, not only in the control of decay, but also including observations on such inseparable matters as the possible imparting of objectionable flavors and odors, and any physiological changes that might occur under its protection.

The Sunkist Growers laboratory continued to test the action of biphenyl in cooperation with paper manufacturers until Adalbert Farkas came to the United States early in 1942. Farkas then personally participated in further tests by the Sunkist workers and the Fernstrom Paper Mills. At this time other agencies, sometimes at the request of Mr. Farkas, began making tests with biphenyl wraps. Active among these agencies was the United States Department of Agriculture at such points as Chicago, New York, Pomona (California), and especially at Orlando (Florida).

The expanding use of the fiberboard carton brought a renewed interest in the action of biphenyl on account of its widespread adoption as a protective agent in connection with packing operations. The use of biphenyl-treated tissue wraps declined in the United States, but in its stead came biphenyl-impregnated "liners," "collars," "pads," etc., to be inserted in the cartons just before filling them with fruit; or the interior surfaces of the cartons might be treated with biphenyl. These modifications of the application of biphenyl

should not be expected to alter significantly the principle involved in its functioning as a mold control. However, the tightness of the nonventilated types of cartons may allow higher or more prolonged build-ups of biphenyl in the atmosphere surrounding the fruit than with standard boxes or biphenyl-treated wraps. This becomes of especial interest, and is in question, when long holding in the warehouse after packing is planned.

The desirability of referring to biphenyl as a fungistat rather than a fungicide was first noted by Farkas and Aman (1940) while studying the action of biphenyl vapor on various molds. Biphenyl in concentrations of 0.08 milligram per liter of air stopped the development of green and blue molds and *Diplodia* completely. However, the spores and older hyphae continued to grow when the biphenyl was removed. That is to say, the fungi had not been killed by the biphenyl, but their growth was held in abeyance, or made static. Since this action of biphenyl was first reported, the same characteristic has been noted by other workers.

Biphenyl is a volatile fungistat which is used chiefly in packaging materials for citrus fruit. Its fungistatic properties are limited mostly to citrus, since it imparts off-flavor and off-odor to most other fruits and to vegetables. Biphenyl has been absorbed on paper wraps, paper sheets, and box liners, but the current customary procedure in the United States is to use 2 Kraft paper sheets with 2 to 3 grams of biphenyl per sheet in each fruit container. This compound, which has a relatively low vapor pressure, evaporates slowly enough to produce an effective concentration in the surrounding air. This results in the prevention of decay and soilage of grapefruit by limiting fungus growth and inhibiting the surface sporulation of blue and green molds.

The citrus industry of South Africa is dependent upon biphenyl for protecting its fruit during nonrefrigerated transit from packing houses to the precooling facilities at shipping ports. It also protects the fruit during the refrigerated period of ocean transportation to export markets, and for a limited period in the early stages of distribution in the importing countries. The South African citrus industry uses more biphenyl per pound (0.454 kilogram) of fruit than is used in Israel, and less than that used in the United States. In a 40-pound carton of fruit (72 oranges), the South African Cooperative Citrus Exchange uses approximately 3.6 grams of biphenyl in 76 tissue wraps, the Citrus Marketing Board of Israel uses 2.1 grams in 76 tissue wraps, while the California-Arizona citrus industry uses no fruit wraps, but inserts 2 paper pads in each carton, each pad being impregnated with approximately 2.25 grams of biphenyl, which amounts to 4.5 grams (2 pads) per box of fruit.

In those areas where fruits are individually wrapped, specifications are usually required for the biphenyl content of standard tissue wraps. In South Africa, the biphenyl content varies from 40 to 60 milligrams per 100 square inches (*ca.* 645 cm^2); in Israel, the biphenyl content ranges from 28 to 32

milligrams per 100 square inches. There is a consistent effort on the part of the citrus industries of both South Africa and Israel to reduce to a minimum the amount of biphenyl used in fruit containers, but it is important to note that the fruit is protected from fungal decay only by the concentration of biphenyl in the atmosphere surrounding the fruit.

Under the auspices of the United States Department of Agriculture, Rygg (1969) directed a series of test shipments of California citrus fruits to European ports for the purpose of determining the biphenyl residues of the fruit on arrival at port, and after a period equivalent to the time required for distribution to the various markets. During these shipments, the transit temperatures and fruit condition were recorded in relation to the various methods of transportation.

The grapefruit data resulting from these experiments showed that the transit temperatures of 5 shipments extended from 47 to 54° F. (8.3 to 12° C.), which was an acceptable range for grapefruit. The transit temperature of test shipment number 6 was 64° F. (17.7° C.), and the fruit in this shipment had high biphenyl residues, a high percentage of decay, and much soilage. The increase in transit temperature resulted in an increase in biphenyl absorption by the fruit, and produced an environment highly favorable for fruit decay.

Because the vapor pressure of biphenyl is a function of temperature, it follows that the amount of biphenyl in the atmosphere surrounding the fruit, as well as the amount of biphenyl residue in the fruit, is correlated with changes in temperature. Consequently, the biphenyl residue of grapefruit, as a result of confinement to enclosures, is chiefly associated with temperature, concentration of biphenyl in the surrounding atmosphere, and the duration of exposure.

The biphenyl residue in grapefruit samples from the prompt and delayed loadings representing the 5 acceptable shipments amounted, on the average, to 38 and 50 ppm, respectively. These amounts were far below the tolerance level of 70 ppm. The principal exception to these values occurred in test shipment number 6, where the average transit temperature was 64° F. (17.7° C.), several degrees higher than the mean values for the other test shipments, and where the transit period was 28 days, or 7 to 8 days longer than the time required for the other shipments. The biphenyl content of the fruit in this particular shipment varied from 64 to 79 ppm. Delayed loading and holding of fruit after arrival apparently did not affect its biphenyl content; furthermore, no measurable decrease occurred during the airing of the fruit.

Schiffmann-Nadel (1969) reported that under certain conditions, the wrapping of grapefruit in biphenyl-impregnated tissues did not always reduce the amount of rot in the fruit to a significant degree. Fruit placed in wraps containing 30 milligrams of biphenyl per 100 square inches (*ca.* 645 cm^2) was variable in the amount of stem-end rot reduced during prolonged storage, but

the data from grapefruit wrapped in tissues containing 60 milligrams per 100 square inches showed considerable reduction in stem-end rot. In one of her experiments in which grapefruit was stored at 10° C. (50° F.) for 4, 8, 12, 16, and 20 weeks, the fruit wrapped in tissues *without biphenyl* had 0, 0.5, 3.1, 4.1, and 7.6 per cent stem-end rot, while fruit wrapped in *biphenyl-impregnated* tissues (30 milligrams per 100 square inches) had 0, 0.5, 5.4, 2.0, and 5.4 per cent rot. When the wrappers contained 60 milligrams per 100 square inches, the fruits had 0, 1, 0, 0.5, and 1.5 per cent stem-end rot, respectively.

Fungus Diseases of Postharvest Grapefruit

THE SUCCESSFUL MARKETING of fresh grapefruit requires that the fruit be handled carefully from the time it is picked to the time it reaches the consumer. The principle of careful handling of postharvest grapefruit is particularly manifested by the relationship of fruit spoilage to surface injury of the peel.

Smoot and Melvin (1961), in studies on the general nature of the fungus spoilage of citrus fruits, determined the effects of the type and severity of peel injury and of fruit maturity on the susceptibility of several kinds of Florida citrus to green-mold infection. Only their results on Marsh Seedless grapefruit will be reported here. Samples were picked at approximately 2-week intervals, from October, 1960, to April, 1961. After washing and drying, the fruit was randomized into lots of 20 for treatment. The samples were injured by puncture wounds only. The injured fruit was immediately inoculated by immersion in an aqueous spore suspension of *Penicillium digitatum* containing approximately 10 million spores per milliliter. The samples were then incubated at 70° F. (21° C.) for 1 week in open boxes. Inspections were made after 4 and 7 days.

The time-course curves in figure 161 show the effect of maturity on the susceptibility of injured Marsh grapefruit to *Penicillium digitatum*. The punctures at 3-millimeter depth resulted in 90 to 100 per cent incidence of decay throughout the sampling period. In fruit with punctures of lesser depth, the rate of infection increased progressively with the season. The slope of the curve for the 2-millimeter depth was similar to the time course of the soluble-solids:acids ratio, showing that in all varieties, fruit decay increased with fruit maturity.

Stem-End Rot. Stem-end rot causes serious losses in grapefruit shipments from Florida, Texas, and the West Indies during years of high rainfall and excessive humidity, but this pathological disorder is of minor importance in semiarid states such as Arizona and California. The term "stem-end rot" ordinarily refers to the fungal decay usually caused by either *Phomopsis citri* or *Diplodia natalensis*. Other rots of the stem end are caused by *Alternaria citri, Colletotrichum gloeosporioides, Dothiorella* sp., and *Fusarium* sp.

Decay-producing organisms are responsible for considerable losses in fresh grapefruit during storage and transit. Both decay and physiological disorders are highly associated with temperature. Fruit rots show an accelerated increase at 70 to 85° F. (21 to 29.4° C.). Some decay organisms are significantly reduced by a lowering of 10 to 20 degrees Fahrenheit in commodity temperature (about 5.5 to 11° C.). Consequently, most decay-producing organisms are inactivated at 32° F. (zero centigrade), but a few of them develop slowly, even at this temperature.

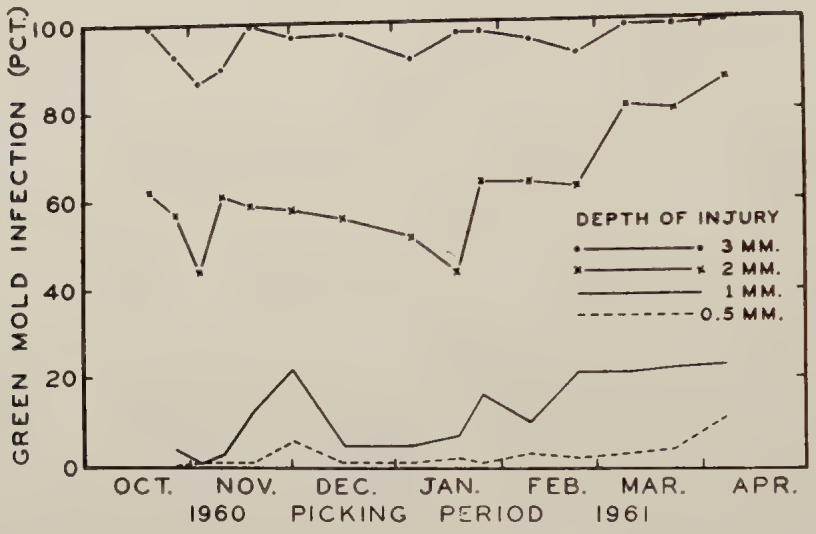


Fig. 161. Effects of puncture injury and fruit maturity on the susceptibility of Marsh grapefruit to green-mold infection. (From Smoot and Melvin, 1961.)

In a 3-year experiment (1964–67) to determine the most suitable storage conditions for Israel-grown Marsh Seedless grapefruit, Schiffmann-Nadel (1969) paid particular attention to the different factors that affected the stem-end rot diseases of refrigerated and nonrefrigerated grapefruit. From the data of figure 162, it can be observed that grapefruit stored at 12° C. (54° F.) had a good general appearance and developed a minimum amount of stem-end rots. In the 2° C. (36° F.) experiments, no measurable stem-end rots developed during the 16 weeks of storage, but at other experimental temperatures, stem-end rots increased at varying degrees with increases in the storage periods. The highest percentages of stem-end rots occurred at storage temperatures of 6° C. (43° F.) and 8° C. (47° F.), amounting to more than 50 per cent of the fruit after 12 and 16 weeks in storage. These temperatures were considerably below those required for the maximum growth of stem-end rot fungi. The percentages of rot at these temperatures exceeded the amount that occurred in fruit stored at 17° C. (63° F.), which temperature approaches the optimum for growth of most stem-end rot fungi. It was reported that at temperatures

above 2° C. (36° F.), the lowest percentages of stem-end rot occurred at 12° C. (54° F.), and the fruit stored at 14° C. (57° F.) contained a slightly higher percentage of rots. Consequently, the best storage results with reference to rots and blemishes were obtained at these 2 temperatures.

At storage temperatures between 4 and 10° C. (39 and 50° F.), stem-end rot was principally caused by *Alternaria citri, Phomopsis citri,* and *Fusarium* sp., but at temperatures between 12 and 17° C., the rot was chiefly caused by *Diplodia natalensis.* During the 2-week holding period at 17° C., stem-end rot continued to develop on fruit stored earlier at temperatures from 2 to 14° C., inclusive. The fungi that were isolated from infected fruit during the 2 weeks of shelf life at 17° C. were the same species that developed during the various storage periods. After prolonged cold storage (16 to 20 weeks) followed by 2 weeks of shelf life, the interior core rot was caused mostly by *Alternaria citri,* and to a much less degree by *Trichoderma viride* and *Fusarium* sp.

In order to obtain further information on this unexpectedly higher incidence of stem-end rot at the lower temperatures of 6 to 10° C. (43 to 50° F.), Schiffmann-Nadel (1969) decided to determine the incubation periods of fungi (*Alternaria, Phomopsis,* and *Fusarium*) causing stem-end rots in grapefruit after inoculation by scratching, and also to observe their rates of growth *in vitro.* The results of these experiments indicated that the growth of the fungi *in vitro* was a linear function of temperatures between 6 and 25° C.

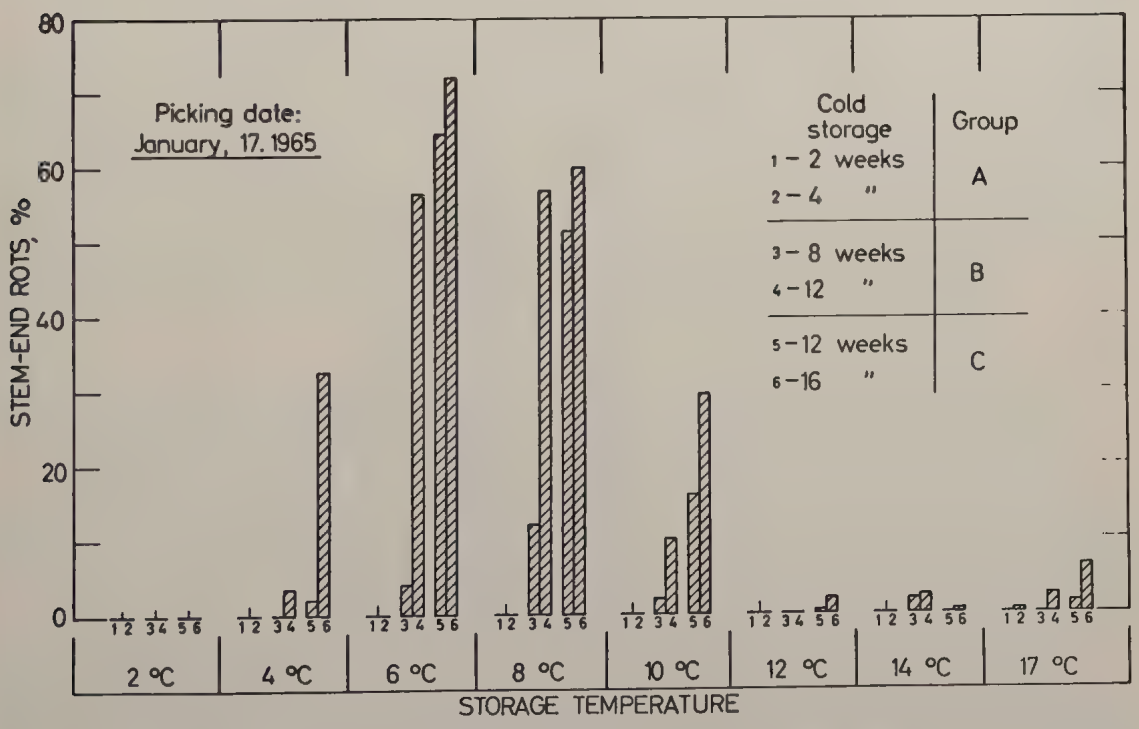


Fig. 162. Effects of cold-storage temperatures on the incidence of stem-end rots on grapefruit wrapped in plain paper after various storage periods. (From Schiffmann-Nadel, 1969.)

(43 and 77° F.), the latter being their optimum growth temperature. As would be predicted, the fruit had the shortest incubation period (between 20 and 30 days) at the optimum growth temperature of 25° C. The increase in the incubation period was inversely related to the temperatures between 20 and 12° C. (68 and 54° F.), and the rate of development of the fungi was decreased. Between 12 and 6° C. (43° F.), the incubation period became significantly shorter, even though the rate of growth of the fungi continued to decrease. Since the fungi had a slow rate of growth between 6 and 12° C., the shorter incubation period at these temperatures might also have been related to direct physiological effects on the grapefruit (chilling injury) as a result of exposure to low temperatures. The fact that considerable rind breakdown occurred on fruit stored below 12° C., especially between 2 and 10° (36 and 50° F.), indicated that grapefruit was affected by these temperatures. See also, chapter 4, page 402.

It is well known that immature oranges and grapefruit are resistant to stem-end rot, and that susceptibility increases with increase in fruit maturity. Minz *et al.* (1947) reported that the incidence of *Diplodia* rot increased with advancing maturity of the fruit. Their data indicated that both the length of the incubation period and the time required for rot to develop were shorter with the more mature fruit. Both oranges and grapefruit became progressively more susceptible to stem-end rot with the approach of ripening.

Heat Conduction of Grapefruit

IN THE REFRIGERATION of citrus fruits in storage rooms or in transit, the 4 thermal factors that must be considered are field heat, vital heat, heat leakage, and internal heat of the conveyance. Precooling of fruit before shipping is closely associated with its refrigeration during transit. "Precooling" is the term applied to the process of rapidly removing the initial heat of the fruit immediately after it has been processed and packed for shipment, so that lower and more uniform temperatures will be maintained in transit without excessive refrigeration.

The heat of grapefruit as indicated by its temperature when picked can be treated as "field heat" or sensible heat. The heat load from this source can be reduced by picking the fruit in the cool of the morning and keeping it out of the sun. The field heat can be calculated by multiplying the specific heat of the load by the weight of the load times the degrees of cooling. (See under the heading "Cooling," that follows closely.)

Citrus fruits, in storage or transit, evolve heat which under adiabatic conditions increases their temperature. This self-heating of the fruit has its origin in the metabolic processes within the fruit, and is commonly known as the heat of respiration. This subject is discussed more fully elsewhere in this chapter; see the section headed "Respiration" on page 447.

Heat leakage and internal heat of the conveyance are 2 factors to be reckoned with in the refrigeration of citrus fruits in transit. Heat leakage is the heat that is transferred from the outside into the refrigerated space, and internal heat is the sensible heat contained in the insulation, inside lining, and inside air of the car or truck before refrigeration begins.

The relatively small amount of refrigeration required to reduce the heat of respiration of citrus fruits and the value of rapidly reducing the field heat of the fruit seem to justify precooling by mechanical refrigeration. The stage of maturity at which the fruit can be picked and the condition in which it arrives at the market depend greatly upon its temperature after harvest, both before and during transit. If the fruits are precooled, it reduces the amount of refrigeration that would ordinarily be required to counteract the heat of respiration if they were loaded at a higher temperature. In addition to reducing the ripening processes during shipment, precooling and refrigeration are effective in reducing the fungal decay of citrus fruits in transit.

Powell (1908) was one of the first to show that refrigeration was effective in retarding the growth of blue and green molds, which he stated would usually enter the fruit through mechanical injuries, adding that the fungal decay would again become active when the fruit was exposed to higher temperatures.

Low-temperature storage is the foundation of quality protection. Reduced temperatures slow the rate of respiratory activity of citrus fruits, reduce water loss, reduce the chance of new decay invasions, and inhibit or reduce the growth of incipient infections. However, low-temperature storage is not a solution to *all* the problems of quality protection. Many tropical and subtropical fruits are subject to injury at temperatures well above their freezing points. If mature-green tomatoes are subject to sustained temperatures below 55° F. (13° C.), they will not ripen fully, and will become highly susceptible to decay.

The same is likewise the case with bananas and many other crops.

Cooling

THE FIELD HEAT of a load of grapefruit may be readily calculated, provided that the specific heat of the fruit is known. It is well understood that equal masses of different substances require unequal quantities of heat to raise their temperatures through the same range. Therefore, the specific heat of a substance is simply the quantity of heat required to raise the temperature of the unit mass of the substance 1 degree. Turrell and Perry (1957) determined the specific heat of Washington Navel oranges, and showed that their specific heat increased in direct proportion to the percentage of water in the fruit (figure 163). Although these determinations were made on oranges, a similar relation should hold for grapefruit.

These authors also determined and compared the rates of cooling, as indicated by changes in temperature, of large (surface area 218 cm^2) and small (surface area 152 cm^2) Marsh grapefruit, in both the center and surface areas of the fruit. The data in figure 164 show the changes in fruit temperature during 150-minute periods. The loss of heat from the centers of the large fruits was at a much slower rate than was the case with the corresponding loss from the centers of the small fruits.

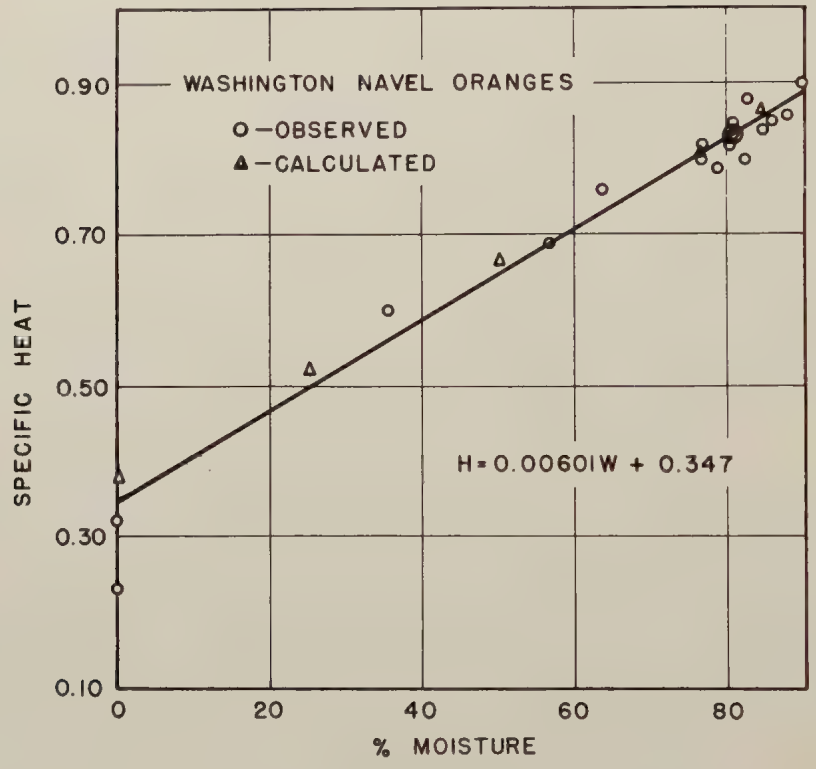
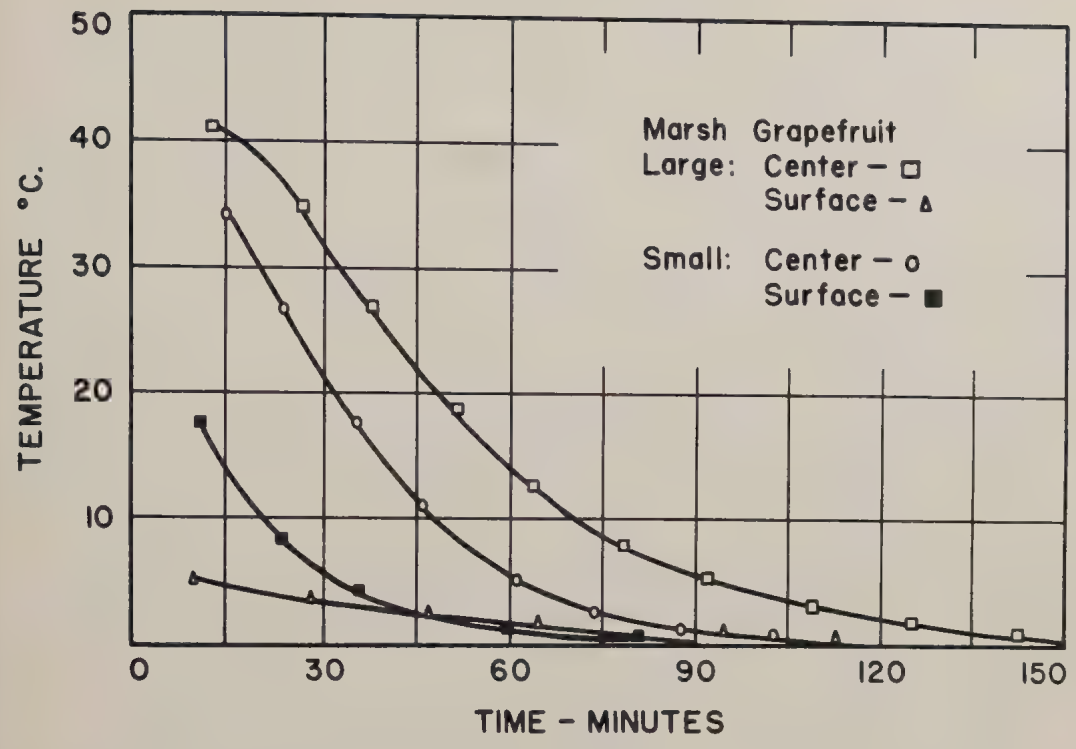
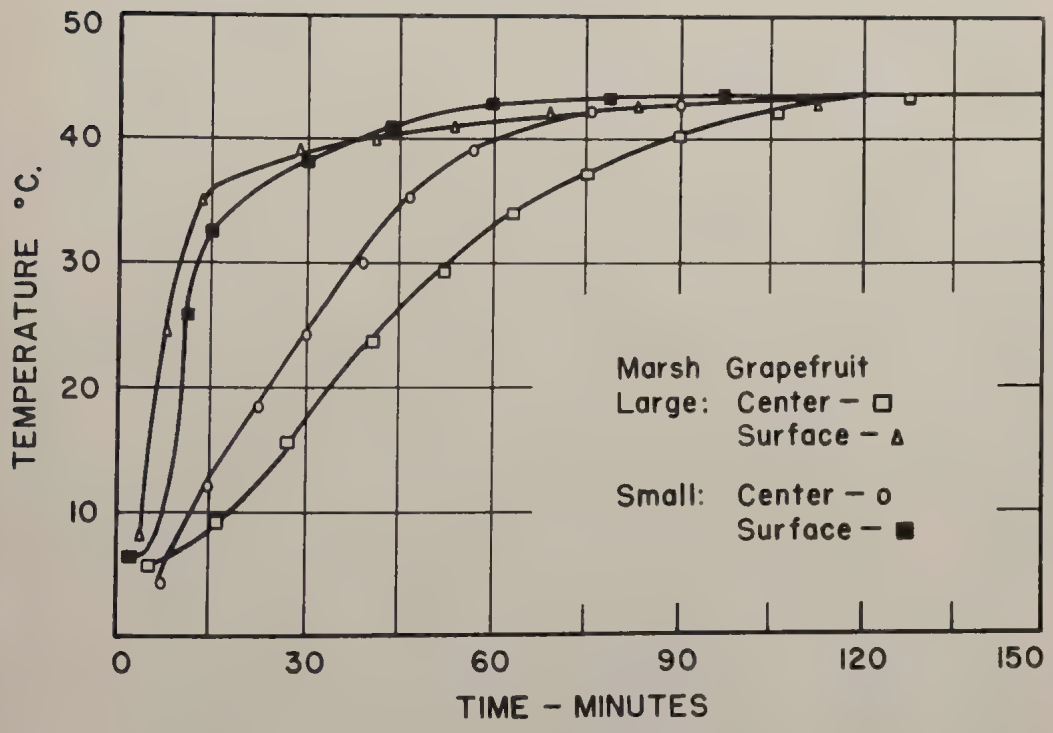


Fig. 163. Regression line of specific heat on the percentages of water content of Washington Navel oranges. (From Turrell and Perry, 1957.)

In still another series, the same authors recorded the heat absorbed, as indicated by temperature, by large and small Marsh grapefruit. The centers of the small fruits reached 40° C. (104° F.) in a shorter time interval than was the case with the large fruits (figure 165). From a practical point of view, the curves shown in figures 164 and 165 would also be significant for fruits attached to the trees in periods of freezing temperatures; under identical conditions, the small fruits would reach an equilibrium temperature with the surrounding atmosphere faster than the large ones.

In Florida, Leggett and Sutton (1951) determined the cooling rates of grapefruit under various experimental conditions. The data in figure 166 show the cooling of grapefruit in cold water, a method commonly known as





Figs. 164 (top) and 165 (bottom). *Top:* Rates of cooling of large and small grapefruit. *Bottom:* Conduction of heat in large and small grapefruit. (From Turrell and Perry, 1957.)

"hydrocooling." The initial temperature of the samples was about 80° F. (27° C.). The cold water in which the crates were immersed had a temperature of approximately 36° F. (2° C.), and the water had a flow of 35 pounds (15.9 kilograms) per minute. The 2 curves show the rates of cooling in crates that were immersed and in crates that were sprayed. After 120 minutes, the immersed fruit had, on the average, a temperature of 44° F. (6.7° C.) and the sprayed fruit, 40° F. (4.4° C.).

Further data of Leggett and Sutton show the air-cooling curves of grapefruit in crates placed at 1-inch (2.5-centimeter) spacing, with the entering air at a temperature of 30° F. (–1.1° C.). The 2 curves given in figure 167 show the rates of cooling at 500 and 1,000 cubic feet (about 14,150 and 28,300 dm^3) per minute per crate, respectively. In the former, the fruit temperatures were reduced to about 40° F. (4.4° C.) in 200 minutes, and in the latter, to the same temperature in 160 minutes.

Similar curves with the crates placed at 7-inch (18-centimeter) spacing are shown in figure 168. The cooling of grapefruit with entering air at a temper-

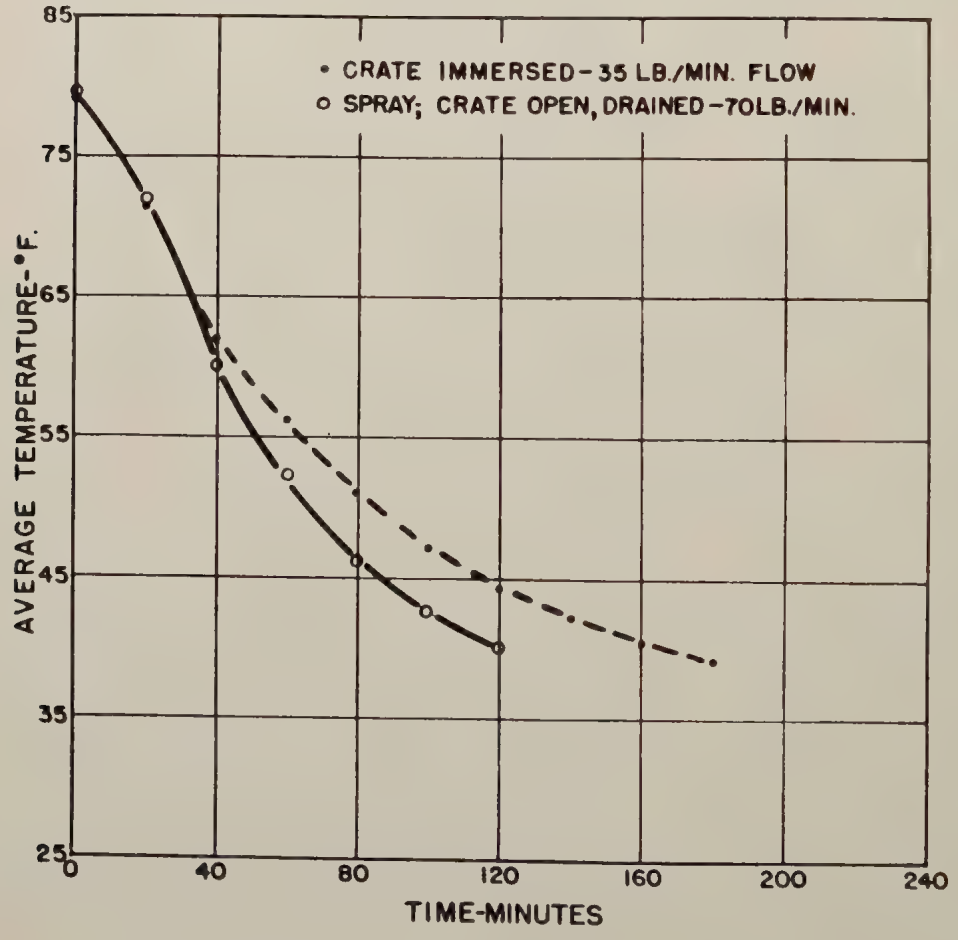


Fig. 166. Cooling curves of grapefruit, using the "hydrocooling" (cold-water) method. (From Leggett and Sutton, 1951.)

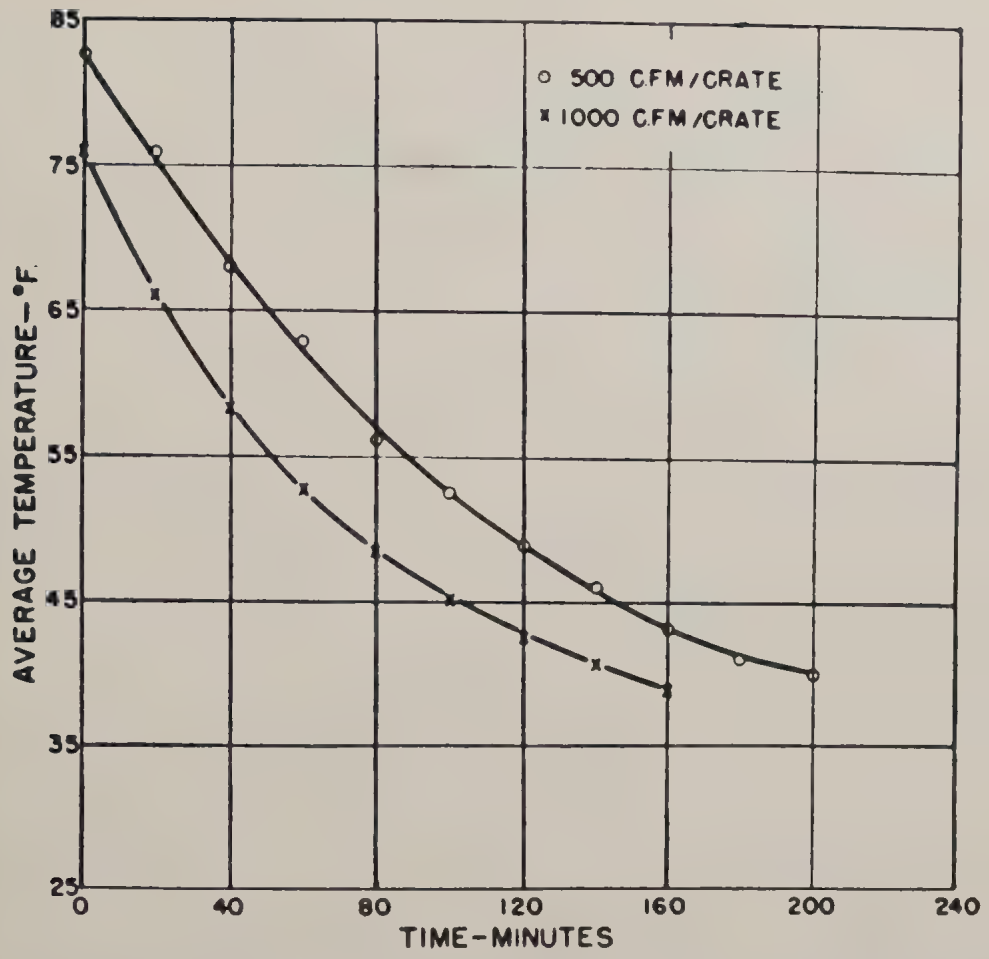


Fig. 167. Cooling curves of grapefruit in crates at 1-inch (2.5-cm) spacing and 30° F. (–1.1° C.) entering air temperature. (From Leggett and Sutton, 1951.)

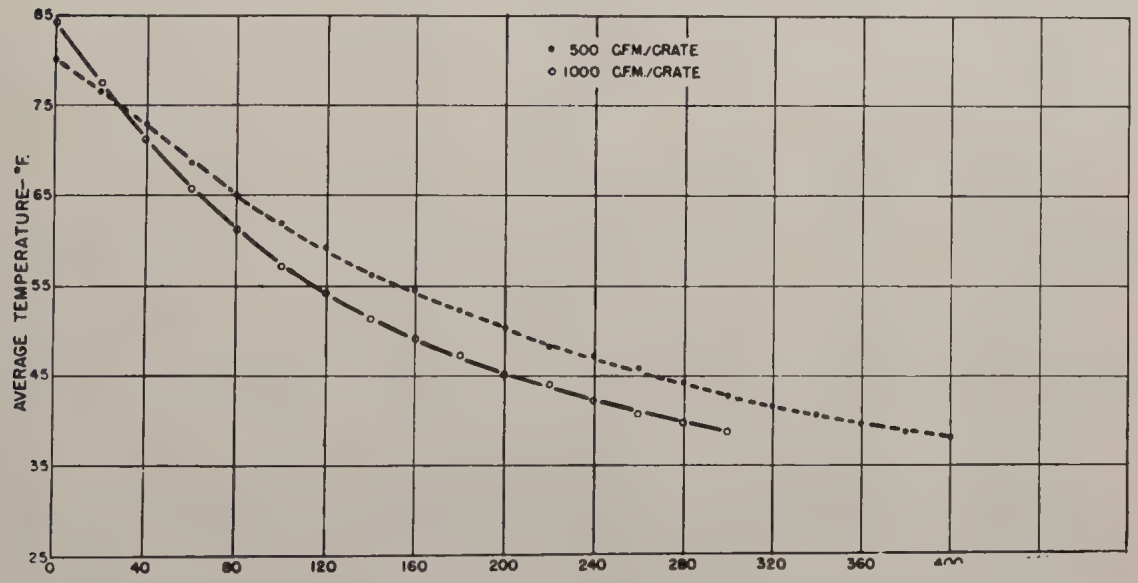


Fig. 168. Cooling curves of grapefruit in crates at 7-inch (18-cm) spacing and 30° F. (–1.1° C.) entering air temperature. (From Leggett and Sutton, 1951.)

ature of 30° F. and with a velocity of air at 500 and 1,000 cubic feet per minute per crate required about 280 and 360 minutes, respectively, to reduce the temperature of the fruit from over 80 to 40° F. (27 to 4.4° C.).

Figure 169 shows the rate of cooling of grapefruit in 1- and 2-layer depths, with air at 30° F. (–1.1° C.), and at velocities of 100 and 200 cubic feet (2,830 and 5,660 dm^3) per minute per square foot (9.29 dm^2). Under these conditions, the temperature of the grapefruit was reduced from 77 to 40° F. (25 to 4.4° C.) in about 130 minutes.

Leggett and Sutton (1951) also determined the coefficient of heat transfer of grapefruit in relation to the quantity of air necessary for cooling the fruit (figure 170).

The coefficient K is the controlling factor governing the rate of heat transfer. As the coefficient K increases, the rate of cooling of the fruit increases, because the difference in temperature between the cooling medium and the body being cooled is decreased.

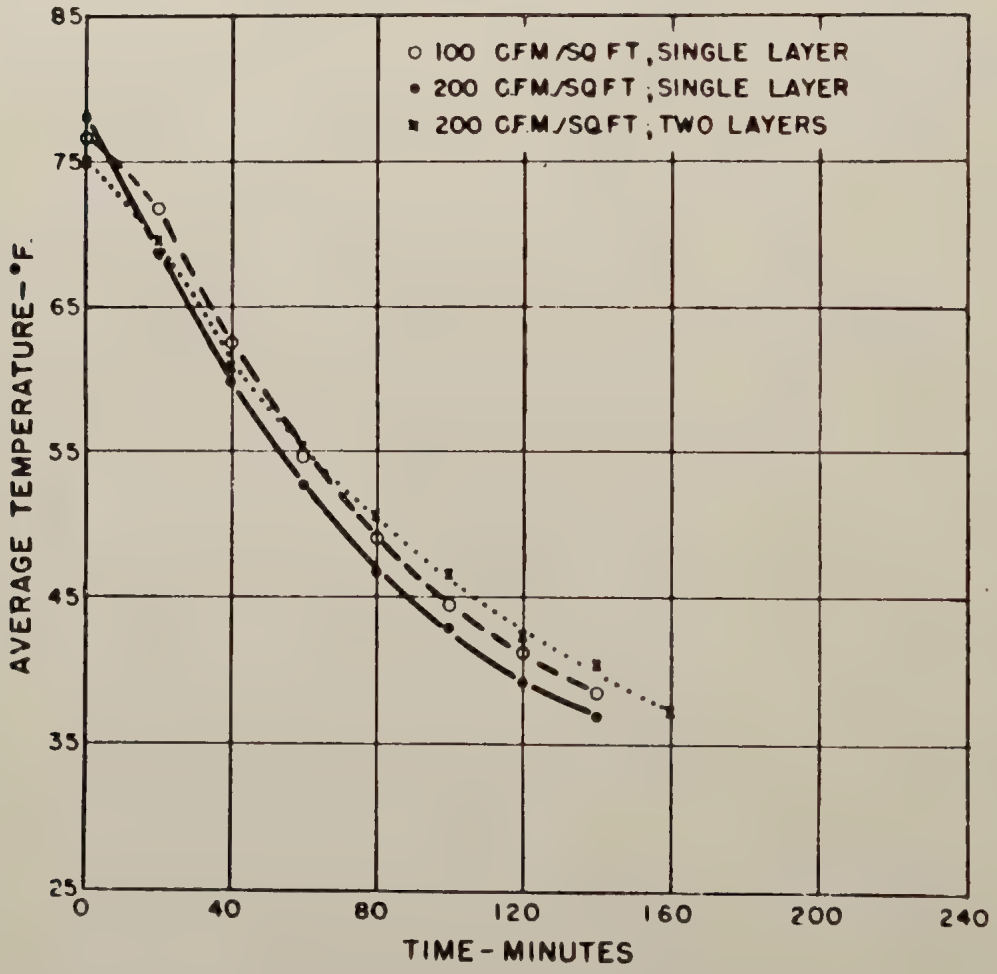


Fig. 169. Cooling curves of grapefruit in layers at 30° F. (–1.1° C.) entering air temperature. (From Leggett and Sutton, 1951.)

Eaks (1954) determined the rate of heat exchange of lemons and oranges packed in standard boxes and in half-box fiberboard cartons. Though he worked with oranges and lemons, his results would, no doubt, apply also to grapefruit. As a result of his tests, Eaks arrived at these conclusions:

In general, the rates of heat exchange were similar for both lemons and oranges, and for fruits at the tops of all containers. The rate of heat exchange was faster (significant at the 1 per cent level) for fruits in the center of the carton than for those in the center of the standard box. The rate of warming was more rapid than the rate of cooling for fruit in the tops of all containers, but not as a rule for fruit in the centers of the containers. Differences in the rate of heat exchange between fruits in the center and outside positions in the standard box were highly significant, whereas in the cartons these differences were small. Venting of the cartons had little or no effect upon the rate of heat exchange in these tests.

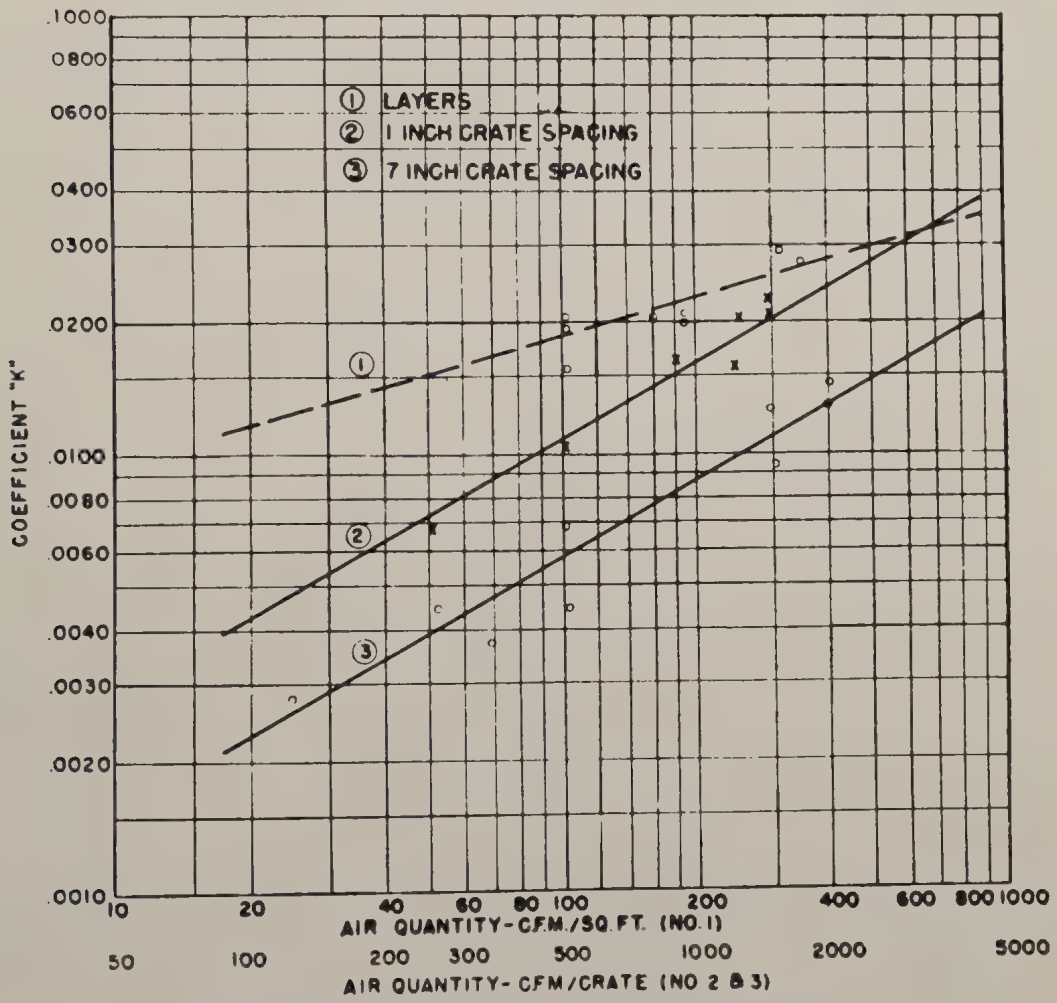


Fig. 170. Relationship of the coefficient K to air quantity for grapefruit. (From Leggett and Sutton, 1951.)

Fruit Pitting

APPARENTLY THE FIRST, or among the first, workers to point out that grapefruit stored at certain temperatures was subject to the physiological malady known as "pitting" were Hawkins and Magness (1920). According to Friend and Bach (1932), pitting—also known as pox, chill-spotting, brown spot, storage spot—is probably the most serious of the cold-storage disorders that attack grapefruit. The disorder consists of abruptly sunken spots in the peel. The spots are not colored at first, but later may become pink, and finally brown. Softening of the tissues under the pits often occurs, and they may become infected with blue mold and other fungal attacks. The pits are small at first, but later may coalesce and become quite large.

Ray Nelson (1933) suggested that pitting is caused by a lack of oxygen. The low temperatures prevent regular respiration, even though there is enough oxygen in the air surrounding the fruit. The less mature the fruit, the greater is the susceptibility to pitting. Ryall and Buford (1947) made the same report.

Harvey and Rygg (1936*c*) studied pitting in grapefruit from 3 localities in California. They stated that fruit from all the localities pitted and spotted most severely at 42° F., least at 52°, and intermediately at 32° (5.5, 11, and 0° C.). The best storage temperature was therefore 52°. In the 3 storage rooms, average relative humidities of 84, 87, and 89 per cent were maintained. These investigators were convinced that if storage temperatures below 42° did not produce excessive spotting of the peel, they tended to increase the susceptibility to spotting on removal to higher temperatures. They also found that fruit which had become pitted while in storage had a relatively higher rate of respiration.

Davies and Boyes (1936), in Capetown, in common with other workers, found that pitting decreased as storage temperatures increased. They reported the following percentages of pitting: 70.6, 50, 8, and 2, respectively, at 37, 40, 45, and 50° F. (2.8, 4.4, 7.2 and 10° C.). Similar results were obtained by Van der Plank *et al.* (1938), also in South Africa, and by Furlong and Barker (1939), who made storage tests on Palestine grapefruit as soon as it arrived in London.

Brooks and McColloch (1936) classified pits as "definite" and "mild." Definite pits were stated to be 5 millimeters or more in diameter, 1 millimeter in depth, and yellow-ocher to ochraceous tawny or tawny in color. The mild pits were simply less pronounced.

Tompkins and Dreyer (1937) stated that: (1) fruit from some trees pitted much worse than fruit from other trees, (2) immature fruit pitted worse than mature fruit, and (3) there was usually more pitting on outside than on inside fruits.

Van der Plank (1938, 1939) reported, as a result of some of his work in South Africa, that small pits about the size of a pinhead formed on the peel surface when grapefruit was stored at 31.5° F. (zero centigrade). These pits were inclined to increase in size when the fruit was exposed to higher temperatures of 35, 39, or 45° F. (1.7, 4, or 7.2° C.).

Rose *et al.* (1943), Miller (1946*a*, *b*), and many others have called attention to the fact that lowering the storage temperature retards, but does not stop, certain of the chemical and physical changes that occur in fruit at higher temperatures. On the other hand, at low temperatures, certain disorders may become pronounced that do not occur at higher temperatures.

For discussions of other physiological and pathological storage and shipping disorders, see Fawcett (1936), Rose *et al.* (1943), Klotz (1961), and Knorr (1965).

Ricey and Coarse-Textured Grapefruit in Relation to Certain Chemical Components. As noted elsewhere, many investigators have studied the seasonal changes in the physical and chemical characteristics of grapefruit during fruit growth and development. In the experiments of Long *et al.* (1959, 1960), they collected packing-house samples of Florida-grown Duncan and Marsh grapefruit, and determined the variations in physical and chemical characteristics with respect to internal quality and their relationship to external quality. Their purpose was to provide a better analysis of these variations, revealing information that would enable shippers to harvest and supply a more uniform product of substantially higher quality. The principal chemical factors considered in the study were the major constituents that would show changes in concentration as a result of physiological changes that occurred during fruit development on the trees and under postharvest environments. They included total soluble solids, total acids, active acidity (pH), solids:acid ratios, and ascorbic acid. The important physical factors included weight of fruit, volume of juice, internal texture, seediness, and quality of late-bloom fruit. The interactions among these physical and chemical components showed some interesting relationships.

Long and his co-workers found that in agreement with most biological systems, the variation of any given constituent among individual grapefruit may show considerable deviation from the mean values, but this situation does not necessarily indicate the absence of certain correlations among many chemical and physical components of the fruit. In relation to the physical components, the volume of juice per fruit is highly correlated not only with fruit weight (in grams) but with fruit diameter (in inches or centimeters). The physical factors of weight, diameter, and volume of juice are interrelated (or partially correlated) with the texture of the flesh.

It was also noted that good-textured fruit of equal diameter usually contained more juice and was heavier than coarse- or ricey-textured fruit. This

TABLE 175

PERCENTAGES OF FALL AND SPRING HARVESTS OF MARSH GRAPEFRUIT HAVING SPECIFIED INTERNAL TEXTURE AND CONDITIONS, CLASSIFIED BY SIZES OF FRUIT, FOR THE CROP SEASONS 1953–54, 1954–55, 1956–57, AND 1957–58.*

Fruit sizes (number per packed box)	Fall harvest			Spring harvest					
	Ricey	Coarse	Good	Ricey	Coarse	Good	Over-ripe	Dry	Late-bloom
96	11	77	12	0	8	71	11	1	9
80	11	83	6	1	7	71	12	0	9
70	20	78	2	1	10	67	13	1	9
64	26	73	1	1	10	71	9	2	7
54	22	78	0	0	14	64	13	1	8
46	34	66	0	1	14	62	15	2	6

*From Long *et al.* (1959).

relationship would not be so apparent in years of high juice volume, but in years of low juice volume, the coarse- or ricey-textured fruit would have more difficulty in passing the juice requirement.

The data in table 175 show the internal texture of fall and spring samples of Marsh grapefruit, classified according to fruit size (Long *et al.*, 1959). The juiciness of the vesicles, which is an important internal quality, is highly associated with what is generally considered as internal texture. Grapefruit which has a ricey- or coarse-textured internal condition has not developed the juice volume necessary to distend the vesicle walls to the thinness found in good-textured fruit. Consequently, the internal texture of early-mature fruit progresses rather rapidly from ricey to coarse to good quality, and with the advance of the season, the fruit begins to desiccate and show signs of overripeness. This was verified by the fact that most of the fruit harvested in the fall was ricey and coarse in texture. The highest quantity of coarse-textured fruit (83 per cent) was found in size 80, and the lowest amount (66 per cent) occurred in size 46. The highest proportion of ricey fruit (34 per cent) occurred in size 46, and the lowest amounts (11 per cent) were observed in sizes 96 and 80. With such large percentages of affected fruit in the fall harvest, the good fruit that remained amounted to 12 per cent of size 96, 6 per cent of size 80, and 2 and 1 per cent in sizes 70 and 64, respectively.

In the samples harvested in the spring, most of the fruit was of good quality, the actual values showing 62 per cent good in size 46, with variations from 64 to 71 per cent in the other sizes being classed as good fruit (table 175). The coarse-textured fruit varied from 7 per cent in size 80 to 14 per cent in sizes 54 and 46. Fruit in an overripe condition ranged from 9 per cent in size 64 to 15 per cent in size 46. Late-bloom fruit accounted for 9 per cent in sizes 96, 80, and 70, ranging down to only 6 per cent in size 46.

[*Turn to page 500.*]

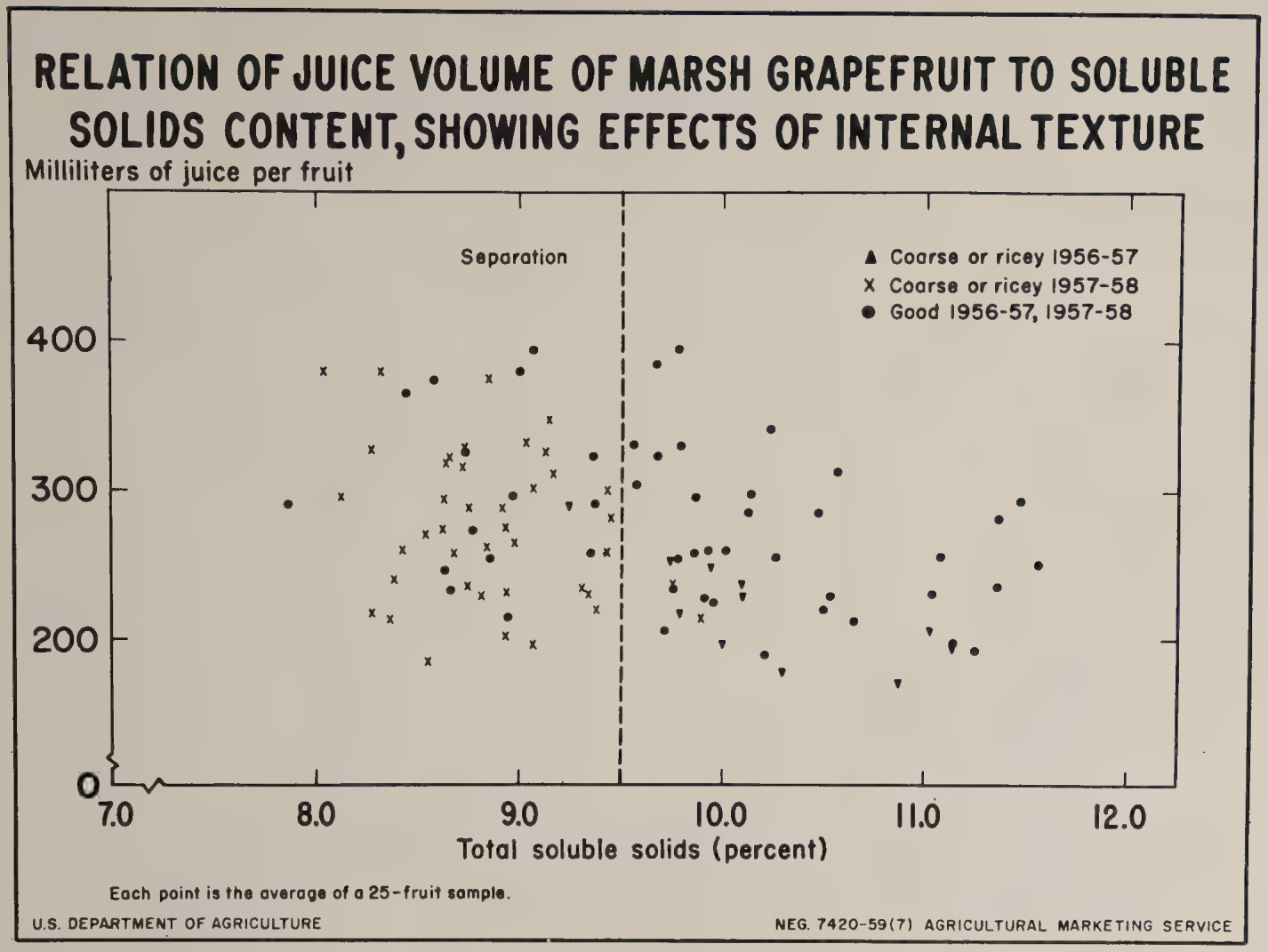


Fig. 171. From Long *et al.*, 1959. (See text for details.)

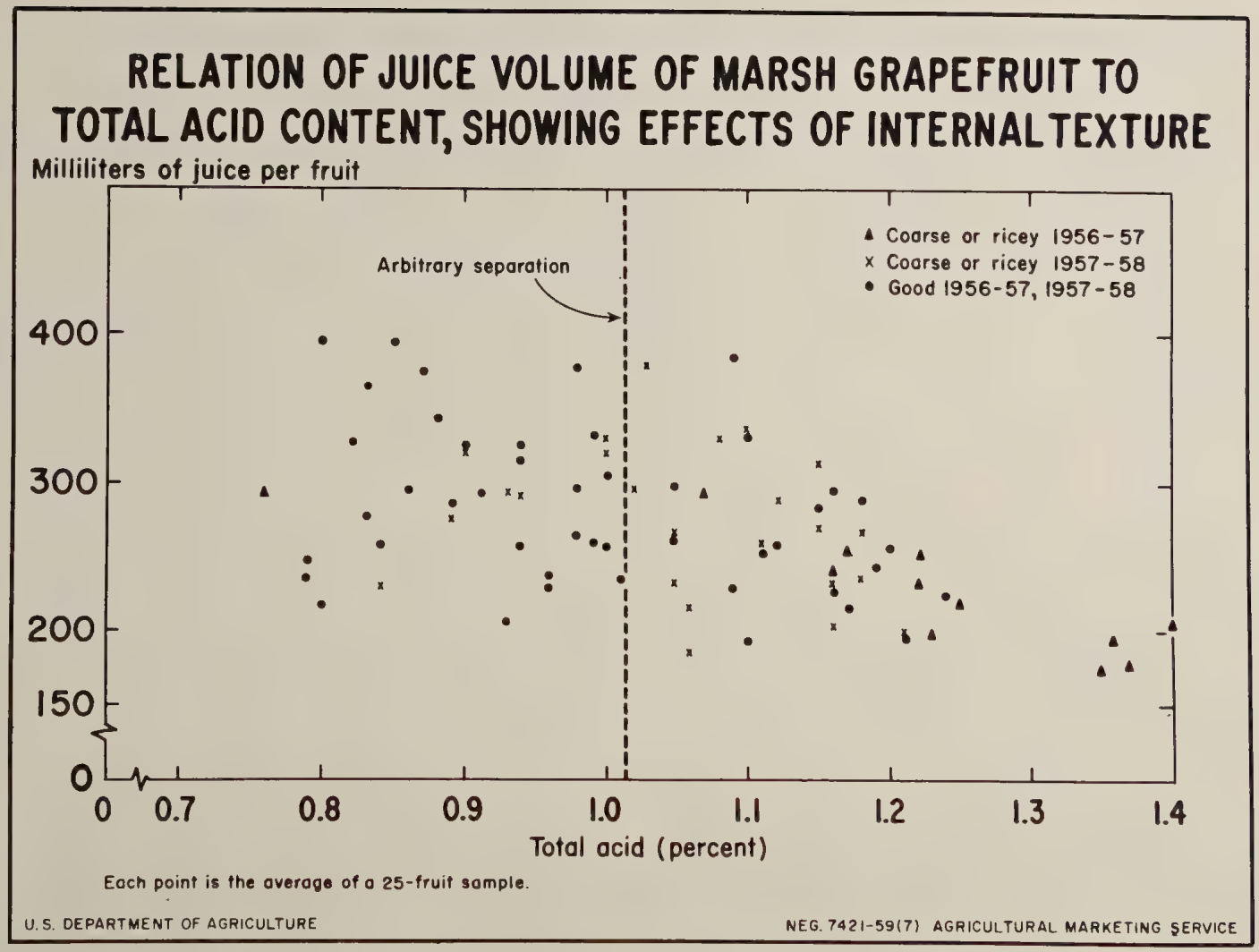

RELATION OF JUICE VOLUME OF MARSH GRAPEFRUIT TO TOTAL ACID CONTENT, SHOWING EFFECTS OF INTERNAL TEXTURE
Milliliters of juice per fruit
Arbitrary separation
▲ Coarse or ricey 1956-57
x Coarse or ricey 1957-58
• Good 1956-57, 1957-58
400
300
200
150
0
0 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4
Total acid (percent)
Each point is the average of a 25-fruit sample.
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Fig. 172. From Long *et al.*, 1959. (See text for details.)

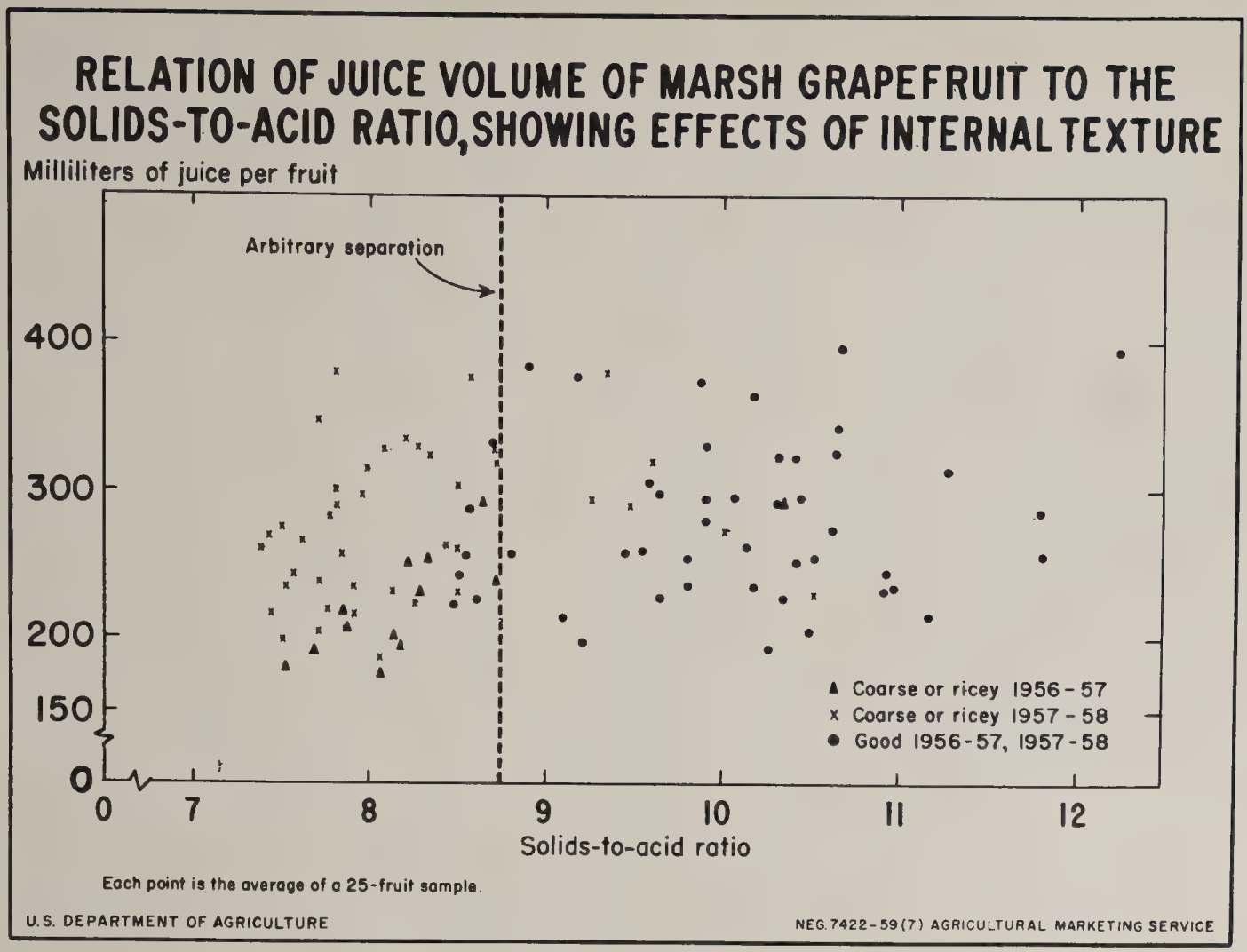

Fig. 173. From Long *et al.*, 1959. (See text for details.)

Since the texture of Marsh grapefruit passes through rapid seasonal changes from coarse to good quality, this particular characteristic should have some degree of association with changes in total soluble solids, total acidity, and solids:acid ratio, because these constituents are correlated with the growth and development of the fruit.

Long *et al.* (1959) plotted the juice volume per fruit against the total soluble solids, and designated those samples which were ricey and coarse and those which were good-textured. A distinct separation between coarse- and good-textured fruit occurred at 9.5 per cent soluble solids (figure 171). In Marsh grapefruit, it appears that 9 to 9.5 per cent soluble solids was found to be the minimum amount necessary in order for it to be considered as good-textured grapefruit.

When the volume of juice (milliliters per fruit) was plotted against the total acid (per cent), a division of the fruit occurred on the abscissa at 1 to 1.02 per cent acid content which arbitrarily separated the fruit having good internal texture from that having ricey- and coarse-textured juice vesicles (figure 172). Although the acid content decreased with fruit growth and development, it also displayed a decided tendency to vary inversely with the juice volume.

In similar experiments where the soluble-solids:acid ratio was plotted against juice volume, the separation of the fruit samples into good- and coarse-textured fruit occurred at a ratio of approximately 8.75:1 (figure 173). The scattering of the values on the graph shows that below the ratio of 8.75:1, 46 fruit samples were coarse and ricey and only 6 samples were of good quality, while above that ratio, 41 samples were of good texture and 7 samples were coarse and ricey. All samples reported on the graph were above the ratio of 7:1, which indicated that all were above the required minimum ratio. In the original report, attention was drawn to the fact that the exceptions in the total acid and solids:acid ratio graphs (figures 172 and 173) were computed on fall samples for 1957–58, a season when the volume of juice and the ratio were found to be higher than average, and the totals of soluble solids and acid were somewhat lower.

In their studies on variations in quality of Florida-grown Duncan grapefruit, Long *et al.* (1960) found that the results resembled their data for Marsh fruit, except that the percentages of total soluble solids and acids were higher, the solids:acid ratio was lower, and the fruit characteristics varied less than those reported for the Marsh variety. When the volume of juice was plotted against the various chemical components, as was done with Marsh grapefruit, separations occurred between the different internal textures of the fruit. When subsample averages for 1955 and 1957 were plotted, the line of separation between fruit with good texture and that with coarse or ricey internal quality occurred at the solids:acid ratio of 8.5:1, the same as in Marsh grape-

fruit. Overripe or dry fruit had lower total soluble solids and acid and lower juice volume, but had higher ratios. Ricey- or coarse-textured fruit had not attained its maximum juice content, but the overripe or dry fruit had lost juice volume by losing moisture through evaporation. The interrelations between juice volume, total soluble solids, acid, and ratio tended to disappear late in the season after the fruit had progressed through prime maturity to an overripe or dry condition. When most of these values for individual fruits were plotted, a trend approaching a line correlation disappeared, especially in late-season fruit. Any time-course curve showing the relationship with one of these chemical components would diminish noticeably in individual fruits harvested late in the season.

Chapter 6

Grapefruit By-Products

THE PROCESSING of grapefruit into segments and juice was initiated in the late 1920's as a means of expanding market outlets for the rapid increase in United States production. The resultant rapid increase in canned and frozen grapefruit products made the disposal of citrus wastes—peel, rag, and seeds—a major problem in the larger citrus-producing areas. A quarter-century ago, McNary (1947) stated that in 1945–46, it was estimated that 1.805 million tons[1] of citrus fruits were processed in Florida canneries. Since the peel, rag, and seeds normally amount to about 57 per cent of the whole fruit, some 1.030 million tons, containing about 82 per cent moisture, were left to be disposed of. In the meantime, however, grapefruit production and processing have remained relatively constant, as shown by Hendrickson and Kesterson (1965) in figure 174. The Florida citrus industry introduced frozen grapefruit concentrate in 1948, and this commodity averaged nearly 1 million gallons[1] a year between 1949 and 1951. Since the peak years, the annual pack has fluctuated considerably, and the production rate has shown no tendency similar to the accelerated rate of growth of frozen orange concentrate. For comparative purposes, it can be observed in the graph that the continued increase in total production of Florida oranges resulted in similar increases in the amount of fruit converted to juice products. Consequently, there resulted a parallel increase in waste residues, which have served as source materials for the manufacture of citrus by-products. In the 1961–62 season, Hendrickson and Kesterson (1965) reported that citrus-peel residues for all varieties amounted to nearly 4.5 billion pounds of peel, rag, and seed which were available for disposal through various means, such as research and the manufacture of citrus by-products (figure 175).

It follows that waste disposal and by-product utilization have grown in importance along with the growth of the food-processing industries. Solid waste from the citrus-processing industries amounts to thousands of tons annually in the United States, and over the years, considerable progress has been made in the economic disposal of both solid and liquid wastes. More efficient filtration and chemical treatments have significantly reduced the

[1] One ton equals 2,000 pounds; one pound equals 0.454 kilogram. One gallon equals 3.79 liters.

nuisances of liquid effluents. In other words, usable products have been recovered from concentrated liquid effluents.

The products or biological complexes that have been obtained or derived from citrus fruit tissues by commercial processes or by pilot-plant procedures are diagrammatically illustrated in figure 176 (Hendrickson and Kesterson, 1965). The chemical constituents that can be isolated from grapefruit and other citrus fruits are obviously the compounds that exist in the fruit as free chemical entities, or in combination with other compounds to form polymers and other structural components of the fruit. This scheme can be expanded to show in considerable detail the possibility of producing, through chemical and physical methods, the individual compounds that make up the composition of these various fruit components. The economic success in the manufacturing of citrus by-products depends upon the selection of those products that have market possibilities. In the instances where the margin of profit between production and sales price is small, it is important that all components of the fruit be recovered and utilized.

A close inspection of the scheme shows that the first step in the processing of citrus fruits is the extraction of the juice, which subsequently permits the separation of the fruit into peel residue, raw juice, seeds, and waste water. All products are derived from these 4 fractions. Since the fruit is processed specifically for the purpose of producing juice products, the peel and mem-

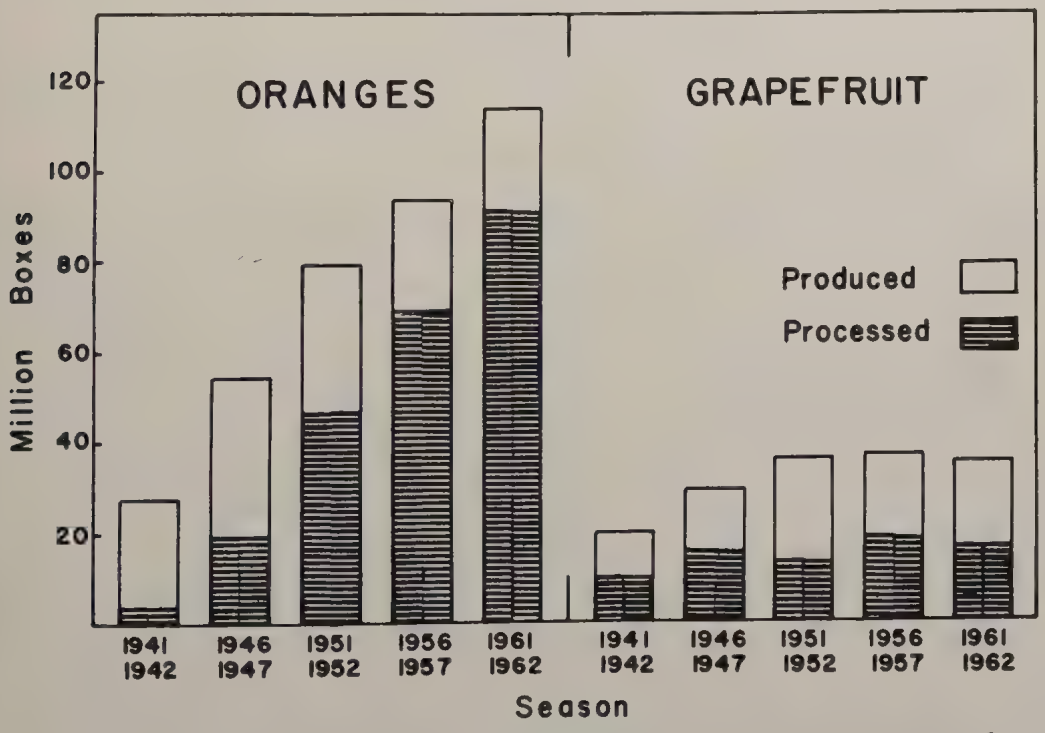

Fig. 174. The quantities of Florida oranges and grapefruit processed, as well as totals available, at 5-year intervals. (From Hendrickson and Kesterson, 1965.)

brane fraction would be expected to serve as a source material for the recovery and production of most of the by-products.

The complex nature of the procedures required for processing citrus fruits is highly associated with their complex morphological structure. Unlike most fruits which are more or less homogeneous, with thin peels and uniform pulp and seeds, citrus fruits are divided into distinct compartments, including thick peels (flavedo and albedo) with oil glands, the locular walls of the segments (carpels), and the pulp residing within the segments, composed

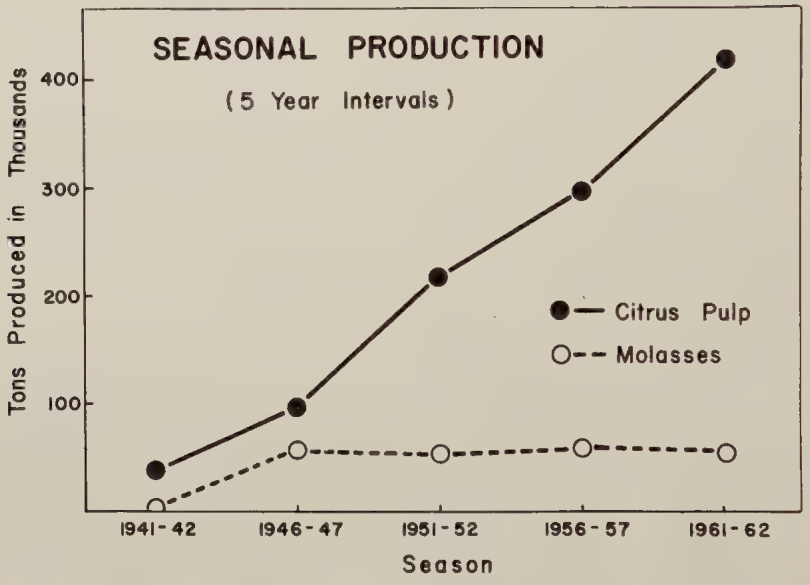

Fig. 175. The production of dried citrus pulp and molasses in Florida, shown at 5-year intervals. (From Hendrickson and Kesterson, 1965.)

of vesicles or sacs which contain the juice. With the highly developed processing techniques adapted to citrus fruits, the peel, pulp, and juice of grapefruit can be efficiently separated. Since the juice is retained in special receptacles, its separation from the other tissues without too much contamination has been highly successful. The general scheme (figure 176) can be considerably expanded with the detailed flow sheets of the production and separation of the essential oils, pectic substances, and other fruit constituents.

The residue that remains after extracting the juice from citrus fruits is the source material for production of dried citrus pulp, which has found a substantial use as a stock feed, and which also serves as the raw material from which many citrus by-products are produced. Citrus molasses is another important general product that is manufactured from the pressed liquor obtained from the lime-treated citrus-peel residue. Molasses is the raw material for the production of citric and lactic acids, vinegar, butylene glycol, and alcoholic beverages.

The citrus residues from the juice extractors can be better understood by considering the flow and total solids balance sheet for the processing of a known volume and weight of grapefruit (figure 177). Hendrickson and Kesterson (1965) extracted the juice from 1,000 boxes of grapefruit (85,000 pounds or 38,590 kilograms of fruit), which amounted to 47.9 per cent of the total weight of the sample. The juice extractors ejected 44,300 pounds (20,112 kilograms) of peel, pulp, and seeds, which was equivalent to 52.1 per cent of the total sample weight. This material contained 81.9 per cent moisture. The peel residue was disintegrated to a suitable size in powerful hammer mills or shredders, and was subsequently treated with lime—CaO or $Ca(OH)_2$—and then thoroughly mixed in a reaction conveyor in order to complete the reaction between lime and the acidic groups of the residue. Before lime treatment, the finely cut peel formed a very sticky and hydrated mass which would be very difficult to dry uniformly without case-hardening. After the lime treatment, the peel residue became easy to press, since the peel mass changed its physical state and exhibited the phenomenon of syneresis, which is the reverse of hydration and imbibition. Consequently, the lime degraded and demethylated the pectic substances in the peel, setting the carboxyl groups free to react with the calcium ions to form insoluble calcium pectates. In the lime-treated mixture, syneresis occurred with the spontaneous separation of the bound liquid containing many soluble peel constituents. The remain-

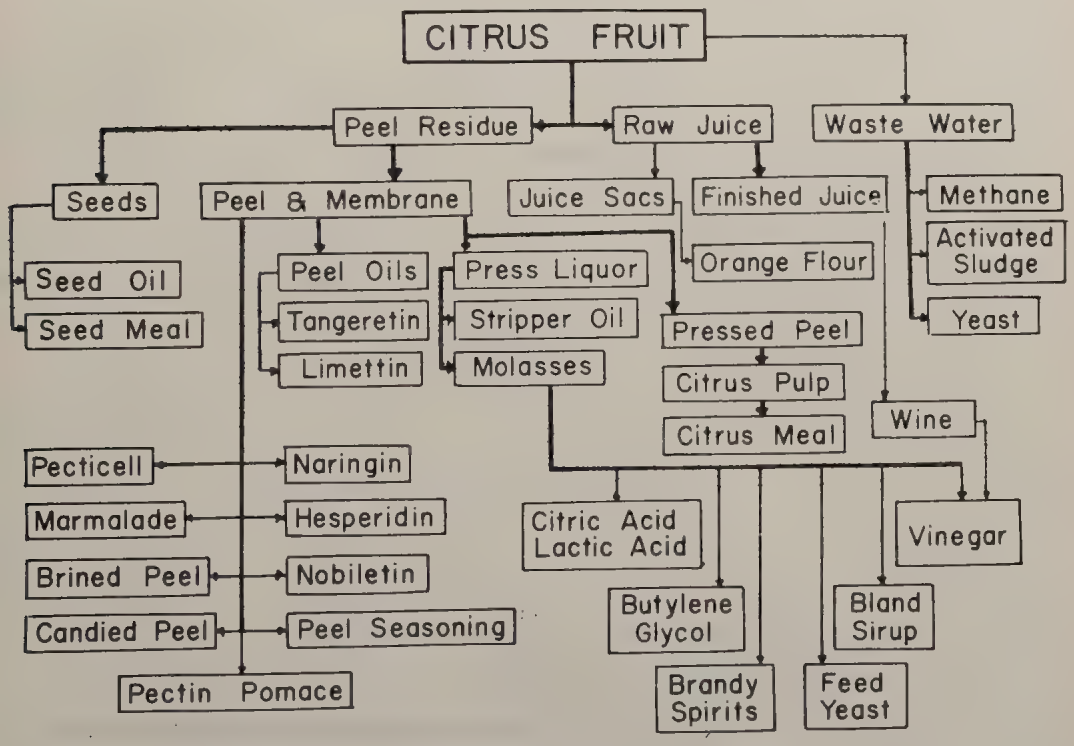

Fig. 176. The origins and interrelationships of citrus by-products. (From Hendrickson and Kesterson, 1965.)

ing mass was no longer sticky, and was easily pressed, either in a screw press or in a frame hydraulic press. It therefore can be observed in figure 177 that the lime-cured peel residue was easily separated into 2 fractions, the press cake and the press liquor. In this particular instance, the press cake amounted to 53.6 per cent, and the press liquor amounted to 46.4 per cent of the total residue ejected by the juice extractors.

As noted in the flow sheet (figure 177), the press cake and press liquor were treated separately from this stage to the final product. The press cake, which contained 76 per cent moisture, was dried in rotary driers to approximately 8 per cent moisture. The dried pulp, the combustion gases (flue gases and oxygen), and moisture were discharged continuously into a cyclone separator, the place where the dry solids were deposited on the walls of the cone and

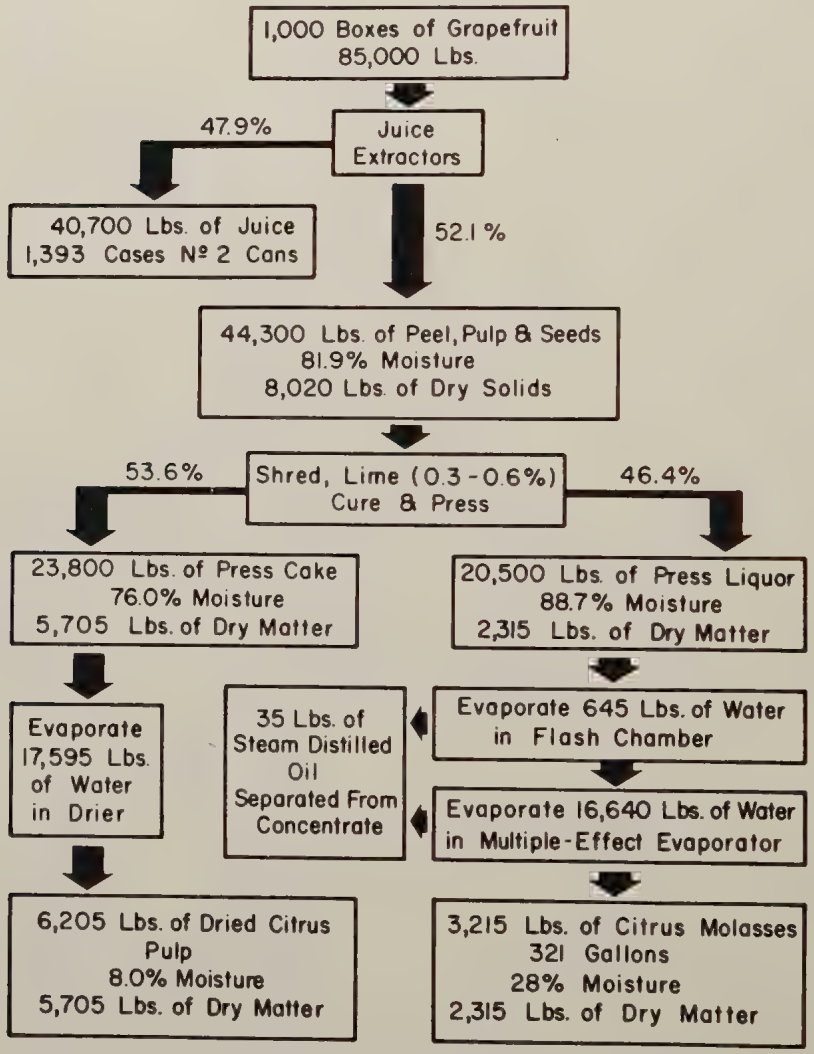
1,000 Boxes of Grapefruit
85,000 Lbs.
47.9%
Juice Extractors
40,700 Lbs. of Juice
1,393 Cases Nº 2 Cans
52.1 %
44,300 Lbs. of Peel, Pulp & Seeds
81.9% Moisture
8,020 Lbs. of Dry Solids
53.6%
Shred, Lime (0.3 - 0.6%) Cure & Press
46.4%
23,800 Lbs. of Press Cake
76.0% Moisture
5,705 Lbs. of Dry Matter
20,500 Lbs. of Press Liquor
88.7% Moisture
2,315 Lbs. of Dry Matter
Evaporate 17,595 Lbs. of Water in Drier
35 Lbs. of Steam Distilled Oil Separated From Concentrate
Evaporate 645 Lbs. of Water in Flash Chamber
Evaporate 16,640 Lbs. of Water in Multiple-Effect Evaporator
6,205 Lbs. of Dried Citrus Pulp
8.0% Moisture
5,705 Lbs. of Dry Matter
3,215 Lbs. of Citrus Molasses
321 Gallons
28% Moisture
2,315 Lbs. of Dry Matter

Fig. 177. Flow and material balance sheet for processing citrus residues into dried pulp and molasses. (From Hendrickson and Kesterson, 1965.)

the hot gases and moisture vapor were discharged into the air. The dried pulp was passed from the separator to a rotating drum which served as a cooler by means of a countercurrent flow of air. This cooling air carried the so-called "fines" or "dust," which were collected separately. A rotary screen attached to the outlet end of the cooling drum separated the remaining peel residue into 2 fractions; one known as citrus meal, and the other as dried citrus pulp. According to Hendrickson and Kesterson (1965), a typical plant in Florida would produce about 85 per cent dried citrus pulp, 14 per cent citrus meal, and 1 per cent fines.

Citrus Molasses

THE PRESS LIQUOR, which may amount to approximately half of the total weight of the lime-cured peel residue, can be concentrated to a thick syrup, commonly known as citrus molasses. Before concentration, this liquor contains from 9 to 15 per cent soluble solids, of which 50 to 70 per cent consists of soluble sugars. Hendrickson and Kesterson (1965) noted that the soluble solids of the press liquor from oranges exceeded those from grapefruit, and the dissolved solids in the peel juice of each species were usually 15 per cent higher than the corresponding pulp juice. Conversely, orange peel had a significantly lower moisture content than grapefruit peel, for which reason it was frequently dried without pressing.

To convert citrus press liquor into molasses, the process is initiated by passing the liquor through vibrating screens of 40- to 80-mesh to remove the suspended solids. The liquor is next superheated to 240–260° F. (116–127° C.) in a heat exchanger, and then flashed into a vapor–liquid separator. The resulting vapors are passed to a surface condenser by which an appreciable quantity of oil is recovered from the condensate as a by-product (see figure 177). The heat treatment not only sterilizes the liquor against spoilage organisms, but precipitates the calcium salts (mostly calcium citrate) that cause scaling on the evaporator tubes. In some instances in Florida, the press liquor is clarified before it is passed to the multiple-effect evaporators. Sedimentation is allowed to occur from the hot-pressed liquor in the storage tanks. The press liquor is concentrated to 50° Brix, and subsequently screened (40-mesh) to eliminate scale particles before concentration to 72° Brix in a single-effect evaporator with forced circulation.

Hull *et al.* (1953) reported that in the production of citrus molasses in California, the press liquor is screened, and then pumped to large-volume settling tanks where sedimentation occurs. After decanting the clarified liquor, it is pumped through a tubular heat exchanger at 190° F. (88° C.), and is then evaporated in a triple-effect long-tube vertical evaporator. The liquid flows upward through 1.25-inch (3.2-centimeter) tubes, and is evaporated with natural convection, except in the third effect, which uses a pump

for forced circulation. The liquid that is discharged from the third effect has approximately 30° Brix. Further concentration of the liquor to 72° Brix (45 to 48 per cent sugar) is performed in a single-effect evaporator with forced-circulation external heat.

In large-scale processing of citrus fruits and in other food-processing industries, the methods used for separating suspended solids of varying degrees of stability from liquids are often dependent upon mechanical processes, such as filtration, screening, centrifuging, flotation, and sedimentation for clarification of the press liquor. Other factors that are important in clarification are the accelerating effects of temperature and pH on the aggregation and precipitation of suspended particles. The insolubles gradually increase with an increase in temperature, and the suspended particles coagulate as the temperature approaches the boiling point of the liquor. During the concentration of the press liquor to molasses, the pH of the liquor decreases by approximately 1 unit. When the press liquor is heated to the boiling point, a reduction in pH of 0.2 takes place.

In his studies on the physical properties of the press liquor of Duncan grapefruit peel, Hendrickson (1950) showed that, as the hydrogen ion concentration changed from pH 3.3 to 7.7, the rate of settling of the suspended particles increased the clarification, during a constant 30-minute period, from 20 to 80 per cent. Flash pasteurization produced changes in the liquor which resulted in reduction of scaling in the evaporator tubes by accelerating the coagulation of the suspended insolubles and by eliminating the buoyance effect of the peel oil. Consequently, clarification before concentration in the evaporators yielded a molasses that was less viscous, more stable, and had higher sugar values and lower ash than molasses obtained from unclarified liquor.

Hendrickson (1950) also stated the following general principles: The press liquor is similar to other biological systems, such as fluids, liquids, and extracts, in which the pH and viscosity values are properties that are highly associated with the hydration and hydrophilic properties of the dissolved materials in solution. The viscosity of a crystalloidal solution in a given solvent is a linear relationship, determined solely by the concentration and temperature at which the measurement is made. In systems which contain lyophilic materials of varying degrees of hydration, the relationship between viscosity and concentration of the dispersed phase takes the form of a parabola. This indicates that the lyophilic materials are solvated; consequently, the particles have varying amounts of the solvent associated with them, and the actual amount of solvent is decreasing with each added increment of dispersed materials. As a general rule, the viscosity of these lyophilic systems decreases with an increase in temperature. In these systems, viscosity changes produced by temperature are associated not only with the viscosity of the

dissolved and dispersed materials, but also with the effect of temperature on solvation. Most lyophilic colloids, such as pectic substances, gelatin, and agar, are more highly solvated at the lower temperatures. The viscosity of partially clarified citrus molasses increases parabolically with the increase in Brix. This type of curve appears to suggest that the sugar and nonsugar solids in the molasses may affect the solvent of the system, resulting in large variations in viscosity values. This phenomenon would be especially observed in nonclarified molasses. As would be expected, the viscosity of citrus molasses decreases with an increase in temperature.

Research and Development. Iranzo and Veldhuis (1948) pointed out that the manufacture of dried pulp and molasses would provide an answer to the problem of disposal of citrus waste—peel, pulp, and seeds. The organic matter would thus be retained and converted into many useful products.

Shearon and Burdick (1948) showed diagrammatically (figure 178) the yield of a ton of Texas grapefruit processed for juice, with the waste converted into molasses and dried-pulp feed: 90 gallons of juice, 179 pounds of dry feed (5 per cent moisture), and 40.8 pounds, or 3.6 gallons, of molasses. (See the footnote on the first page of this chapter for metric conversions.) As can be seen in their "solids balance" calculation, these products accounted for all the solids—soluble and insoluble—in fresh grapefruit. Hendrickson and Kesterson (1965), in their flow and material balance sheet, illustrated in more detail the processing of citrus residues into dried pulp and molasses from Florida grapefruit (figure 177).

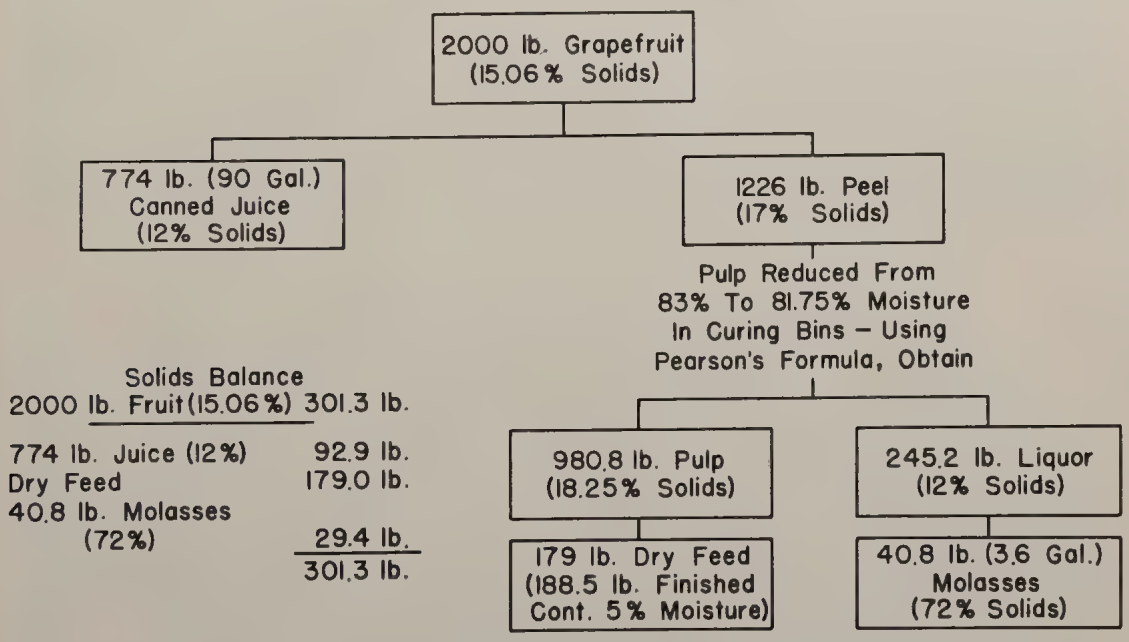

Fig. 178. Simplified diagram of flow and material balance.
(Adapted from Shearon and Burdick, 1948.)

Becker *et al.* (1946*a*) reported that citrus molasses was first produced in Florida during the 1941–42 canning season, and was made from grapefruit pulp. About 2,500 tons were made that season, and 4,700, 10,000, and 24,000 tons during the 3 following seasons. They noted that citrus molasses was used by the mixed-stockfeed trade and for making alcohol, and that the potential production was 1 ton of molasses from 2 tons of dried pulp. Citrus molasses would contain about 57 per cent total digestible nutrients, since it was found to contain approximately 70 per cent dry matter.

Iranzo and Veldhuis (1948) and Cunha *et al.* (1950) stated that in the 1947–48 season, about 66,000 tons of citrus molasses were produced in Florida alone. Becker and Arnold (1951) reported that in 1950–51, this amount had increased to over 70,000 tons.

Ralph L. Miller (1949) reported that during 1948, the dried citrus pulp produced in Florida was worth 4 million dollars, together with 1 million dollars' worth of molasses. If all this pulp and molasses had been fed to cattle, it would have resulted in 15 million dollars' worth of beef at the current market prices.

Shuler and Townsend (1952, 1953) quoted the Florida Canners' Association as stating the tonnages of citrus molasses produced in Florida in the seasons from 1945–46 through 1952–53; their figures follow.

1945–46	44,000	1949–50	42,000
1946–47	58,000	1950–51	70,000
1947–48	66,000	1951–52	54,000
1948–49	41,000	1952–53	39,000

By referring back to figure 175, it will be noted that the seasonal production of citrus molasses, on a 5-year-interval basis for 20 years, remained relatively constant after 1946 (Hendrickson and Kesterson, 1965).

Composition and Feeding Value. Iranzo and Veldhuis (1948) found the composition of citrus molasses to be as shown below. Each value represents the mean of 13 determinations.

Degrees Brix (by refractive index)	71.37
pH	4.68
(The following values are all percentages.)	
Total soluble solids	70.48
Reducing sugars	22.44
Nonreducing sugars	19.65
Total sugars	42.09
Proteins	3.81
Pectin (alcohol precipitate)	1.07
Total acids, as citric	0.64
Volatile acidity, as citric acid	0.53
Total ash	4.77
Calcium in ash	29.84
Magnesium	2.14

Baker (1950) found that citrus molasses contained about 53 per cent digestible nutrients and had about 79 per cent as much nutritive value as snapped corn. As a result of his tests, he concluded that where molasses replaced half of the ground snapped corn ration, the steers did not lose their existing rates of gains in weight or coat finish, had better appetites, were easier to keep on feed, consumed more peanut hay, and had a larger intake of dry matter.

The values shown in table 176 were obtained by Hendrickson (1950) for molasses made from the clarified press liquor of grapefruit peel. The best molasses was made from clarified liquor. Of the 5 methods (filtration, screening, flotation, centrifuging, and sedimentation) used for clarifying the juice, sedimentation proved to be the best. Minute oil droplets tended to retard sedimentation; therefore, it was thought best to remove at least part of the oil by first distilling the liquor.

Swine growers were anxious to use molasses as a feed because it was cheap as compared with corn. The results of previous tests in this field had been variable. Cunha *et al.* (1950) accordingly undertook a study of the problem. They concluded that molasses made from citrus waste could be used successfully for fattening swine when replacing up to 40 per cent of the corn ration. It took from 3 to 7 days for the swine to get used to the taste of citrus molasses.

McClymont (1951) pointed out that citrus molasses was a very poor source of protein; therefore, when used as a stock feed, it should be supplemented by other substances high in protein, such as alfalfa, clover hay, pea-vine hay, linseed meal, or peanut meal. Nevertheless, both citrus pulp and molasses are known to be highly desirable stock feeds. Ralph L. Miller (1949) considered them to be, per unit of total digestible nutrients, the most economical feed ingredients produced at that time. Table 177 shows the values he gave for citrus, sugar-cane, and sugar-beet molasses.

Citrus molasses, as now manufactured in Florida, is required to meet minimum state standards. It must contain 45 per cent total sugars, expressed as

TABLE 176

CARBOHYDRATE ANALYSES OF MOLASSES MADE FROM THE CLARIFIED PRESS LIQUOR OF GRAPEFRUIT PEEL.*

Varieties	Invert sugar (per cent)	Total sugars, as invert (per cent)	Brix, refractive index (28° C. or 82° F.)	Ash (per cent)
Marsh (maximum)....	17.6	51.3	78.4	4.39
Marsh (minimum).....	14.6	44.5	70.3	3.26
Duncan (maximum)...	25.2	56.1	77.7	2.76
Duncan (minimum)...	22.7	50.6	70.7	2.38

*Adapted from Hendrickson (1950).

invert sugar, and test not less than 35.5° Brix by double dilution. A typical analysis is shown in table 178, which gives the averages of values obtained by Iranzo and Veldhuis (1948), Ralph L. Miller (1949), and Hendrickson and Kesterson (1951, 1965).

Millar (1941, 1942) demonstrated that treatment of air-dried sugar-beet pulp with anhydrous ammonia resulted in the fixation of nitrogen in the

TABLE 177

SOME COMPARATIVE VALUES OF MOLASSES DERIVED FROM VARIOUS SOURCES.*

Factors	Composition of molasses from 3 sources (per cent)		
	Citrus†	Sugar cane‡	Sugar beet
Moisture	27.0	25.9	19.4
Degrees Brix	73.0	79.0	79.0
Digestible crude protein	1.4	0.9	2.5
Fat	0.0	0.0	0.0
Nitrogen-free extract	60.0	61.9	62.6
Total digestible nutrients	56.7	56.6	58.8
Dry matter	69.9	74.1	80.6
Ash	5.0	9.4	10.3

*From Miller (1949).
†From Florida Agricultural Experiment Station and Plymouth Research Laboratory.
‡From Morrison (1936).

TABLE 178

A TYPICAL ANALYSIS OF FLORIDA CITRUS MOLASSES.
(Consists of averages derived from 4 sources.)*

Degrees Brix	72.0
Sucrose (per cent)	20.5
Reducing sugars (per cent)	23.5
Total sugars (per cent)	44.0
Moisture (per cent)	29.0
Protein (N × 6.25) (per cent)	4.1
Nitrogen-free extract (per cent)	62.0
Fat (per cent)	0.2
Fiber (per cent)	0.0
Ash (per cent)	4.7
Glucosides (per cent)	3.0
Pentosans (per cent)	1.6
Pectin (per cent)	1.0
Volatile acids (per cent)	0.04
pH	5.0
Potassium (K) (per cent)	1.1
Calcium (Ca) (per cent)	0.8
Sodium (Na) (per cent)	.3
Magnesium (Mg) (per cent)	.1
Iron (Fe) (per cent)	.04
Phosphorus (P) (per cent)	.06
Manganese (Mn) (per cent)	.002
Copper (Cu) (per cent)	.003
Silica (SiO_2) (per cent)	.04
Sulfur (S) (per cent)	.17
Boron (B) (per cent)	.0006
Niacin (ppm)	35
Riboflavin (ppm)	11
Pantothenic acid (ppm)	10
Viscosity at 25° C. (77° F.) (centipoises)	2,000

*From Iranzo and Veldhuis (1948); Miller (1949); and Hendrickson and Kesterson (1951, 1965).

pulp. The amount of nitrogen fixed increased with ammoniation temperature, and a product containing over 10 per cent total nitrogen was obtained. The color of the pulp became progressively darker with increase in ammoniation temperature, but most of the added nitrogen remained in the water-soluble form. The moisture content of the pulp when ammoniated apparently had no effect on the amount of total water-insoluble nitrogen added under these experimental conditions.

Following up these investigations on sugar beets, Stiles (1952) received U. S. Patent No. 2,603,567, which related to a feed supplement for ruminants derived from ammonia- and sugar-containing materials such as molasses. In his patent, he gave the following information concerning the ammoniation of citrus molasses.

> Continuous ammoniation of citrus molasses was effected by pumping a reaction mixture consisting of 13.5 pounds of NH_3 and 538 pounds of citrus molasses at the rate of 30.3 pounds of reaction mixture per hour and at a pressure of 125 pounds per square inch through 180 feet of coiled pipe less than one-half inch in diameter, and heated to 120° C. The residence time of the reaction mixture in the reactor was 13.7 minutes. The product from the reactor contained 3.12 per cent total N and 0.81 per cent free NH_3.[2]

Dried Citrus Feeds

DRIED CITRUS BY-PRODUCTS were suggested as useful for livestock feeding by F. A. McDermott (1917) as quoted by Neal *et al.* (1935*b*).

Probably the first American data on the feeding value of *cooked* citrus waste were obtained and published by Allen (1919). This work was done at the University of Maryland at the request of the Agricultural Commission of the National Research Council. However, it was not until 1926 that dried grapefruit cannery refuse was used for livestock feed (Scott, 1926).

Becker and Arnold (1951) stated that dried citrus pulp first came onto the Florida market in commercial quantities in 1932. From this beginning, the amount of dried pulp produced in Florida increased to 187,545 tons in the 1950–51 season (Florida Citrus Commission, 1951). In the same season, 70,357 tons of molasses were produced. In a later report, the amounts produced in 1952–53 were given as 223,000 and 39,000 tons, respectively.

Hendrickson and Kesterson (1951) stated that approximately 1 million tons of dried citrus pulp had been produced in Florida alone during the 25 years preceding 1951.

Florida, Texas, and California are the only areas in the United States where enough citrus is produced to make it profitable to install machinery for dry-

[2] Metric and temperature conversions for Stiles' patent information: 1 pound equals 0.454 kilogram. Hence, 13.5 pounds equal 6.13 kilograms; 538 pounds equal 244.25 kilograms; 30.3 pounds equal 13.76 kilograms; 125 pounds equal 56.75 kilograms; 1 square inch equals 6.45 square centimeters; 180 feet equal about 55 meters; one-half inch equals 13 millimeters. 120° C. equals 248° F.

ing citrus waste. In some citrus-processing areas, the limed peel is not pressed, but dry pulp is often added to the hydrated peel to prevent agglomeration in the driers. Many processors add various percentages of citrus molasses to citrus feed pulp to raise the carbohydrate content and reduce the crude fiber.

Composition and Feeding Value. As would be expected, tissues that have been dried or partially dried, such as those used for making stock feed, contain a much higher percentage of protein than fresh tissues. Considerable research has been done on the composition and feeding values of dried citrus pulp in order to assess its value as a food supplement for cattle.

Scott (1926) made the following percentage analyses of dried grapefruit waste that was being fed to dairy cows: moisture, 18; fat, 5.25; protein, 5.31; carbohydrates, 61.69; and fiber, 9.75. From his feeding tests, he concluded that dairy cows fed on dried grapefruit waste for 1 year gave increased amounts of milk.

As stated at the beginning of this chapter, the citrus waste from which stock feed is made consists of peel, rag, and seeds. The latter may be practically absent if "seedless" varieties have been used. In table 87, chapter 3, Poore (1934) gave analytical values of fresh California Marsh grapefruit peel and rag combined, and sterilized and stored Florida Marsh grapefruit peel and rag separately. These values for substances in fresh waste may be compared with those for similar substances in dried waste. (*See page 201.*)

Thomas (1934), as well as Neal *et al.* (1934; 1935*a, b*), found that the moisture content of cannery waste (peel, rag, and seeds) was about 80 per cent. In practice, this is artificially reduced to 8 or 10 per cent before it can become a marketable product. It is then placed in bags and allowed to remain in the warehouse, where it becomes air-dry. Dry grapefruit feed is in the class

TABLE 179

COMPOSITION AND FEEDING VALUE OF DRY GRAPEFRUIT WASTE.*

Constituents	Chemical analysis (per cent of waste)	Coefficients of digestibility	Digestible nutrients (per cent of dry matter)
Water	8.23	.....	.....
Total crude protein	4.94	24.83	1.23
Crude fiber	11.94	71.52	8.54
Nitrogen-free extract	69.60	92.43	64.33
Ether extract	1.06	79.37	0.84
Ash	4.23	.....	.....
Totals	100.00	.....	74.94

*Adapted from Neal *et al.* (1934).

of high-carbohydrate concentrates (table 179). When fresh waste was fed to dairy cows, the oil in the peel gave an off-flavor to the milk, but the milk production was increased. Naringin and acid did not keep the cattle from consuming the dry refuse when fed as a part of the otherwise regular ration.

Pico and Ramírez (1935), in Puerto Rico, reported that "bran" made from grapefruit peel had the following percentage compositions: moisture, 7.53; carbohydrates, 78.65; fat, 1.09; protein, 4.94; fiber, 4.82; and ash, 4.97. This product was found to be very palatable to dairy cattle.

Morrison (1936) showed that dried grapefruit, orange, and sugar-beet wastes were very similar in composition. The grapefruit waste was found to be somewhat lower in digestible protein than the other feeds, but slightly higher in nitrogen-free extract.

Table 180 gives the composition of dried citrus stock feed in Florida, as reported by Pulley and von Loesecke (1940). These authors pointed out that the method of drying affected the composition of the feed. Compare the values in the 2 columns of table 180; one gives averages of direct-fire dried and steam-tube dried samples; the other, those from material dried in an

TABLE 180

COMPOSITION OF DRIED GRAPEFRUIT WASTE.*

Constituents	Percentages of dry weight	
	Column 1†	Column 2‡
Moisture	7.54	6.04
Ash	6.50	4.28
Protein (N × 6.25)	7.04	5.70
Crude fat (ether extract)	5.93	3.81
Crude fiber	15.28	10.10
Pentosans	15.57	10.84
Nonreducing sugars (as sucrose)	9.95	25.10
Reducing sugars (as invert)	3.06	7.98
Total sugars	13.01	33.08
Pectin (alcohol ppt)	19.70	17.93
Naringin	1.64	4.10
Potassium and sodium chlorides	1.94	2.67
Other chlorides (Cl_2)	0.05	0.046
Silica	0.25	0.03
Iron and aluminum oxides (Fe_2O_3 and Al_2O_3)	2.10	1.51
Magnesium	0.37	0.12
Calcium	1.77	0.52
Phosphates (P_2O_5)	0.28	0.22
Sulfur	0.11	0.053

*From Pulley and von Loesecke (1940).
†Average of 5 samples, 3 direct-fire dried and 2 steam-tube dried.
‡Material dried in an atmospheric drum drier.

atmospheric drum drier. These authors also reported that waste, limed (1 to 2 per cent) and pressed before drying, had only 33 to 40 per cent as high a sugar content as that dried without pressing, and over 50 per cent more crude fiber.

Chace *et al.* (1940) showed that on a moisture-free basis, grapefruit waste had the following percentage values as stock feed: crude protein, 5.3; crude fiber, 13; ether extract, 1.2; nitrogen-free extract, 75.9; and ash, 4.5.

The following values for the composition of dried grapefruit waste, expressed as percentages of dry weight, were reported by Arnold *et al.* (1941): moisture, 11.3; crude protein, 6.4; crude fat, 5.5; crude fiber, 12.1; nitrogen-free extract, 56.9; calcium, 2.34; magnesium, 0.12; and phosphorus, 0.12. The values were the means from a 3-year feeding test. They found that the average density of dried grapefruit waste was 0.71 pound per quart, and that of dried grapefruit meal, 0.97 pound per quart.[3] The dried waste contained 76 per cent total digestible nutrients. As a result of their feeding trials, they concluded that dried citrus waste was palatable to dairy cows, gave no off-flavor to the milk, and was a desirable bulky carbohydrate concentrate for their rations; also, that the cows produced slightly more milk and butterfat when fed dried grapefruit waste.

Lavigne (1940) also reported that dairy cows produced slightly more milk and butterfat when their feeding ration contained enough dried grapefruit waste to supply 40 per cent of the digestible nutrients. No off-flavor was noted in the milk.

According to Van Antwerpen (1941), dried citrus waste feed will absorb about 5 to 8 times its own weight of water, and thus is valuable in maintaining sufficient water for the bovine diet. The feed must be guaranteed to contain not less than 6 per cent crude protein, 2.5 per cent crude fat, and not more than 20 per cent crude fiber.

Jones *et al.* (1942) stated that Texas cattlemen had begun to use dried citrus waste for feed in 1937. They found that steers fed 25 per cent citrus waste plus 75 per cent corn-ear chop (with husks) gained in weight and finish practically the same as when fed on corn alone. The finish was slightly better when the citrus waste was used. When the feed contained 59 per cent waste, the weight and finish decreased, probably because the feed was less palatable and more laxative.

Copeland and Shephardson (1944) tested the relative effects of 2 kinds of feed concentrates on lactating cows. One feed contained 50 per cent dried citrus waste (about 85 per cent grapefruit and 15 per cent orange); the other, 50 per cent ground corn and cob meal without husks. The remainder of the feed in both cases consisted of 25 pounds of cottonseed meal, 10 pounds of ground oats, 12 pounds of wheat bran, 2 pounds of oystershell flour, and 1

[3] One pound equals 0.454 kilogram. One quart equals 1.10 liter (dry); 0.946 liter (liquid).

pound of salt, a total of 50 pounds. Results: (1) no noticeable difference in flavor or aroma of milk, (2) dried citrus waste was a satisfactory carbonaceous feed for milk cows when used as 50 per cent of the concentrated feed mixture, and (3) increases in milk production and live weight were slightly greater on the ground corn than on the citrus-waste ration.

Shearon and Burdick (1948) made the following comparisons of the feed values of Florida and Texas (Texsun) dried citrus wastes, in percentages.

	Crude protein	*Crude fat*	*Crude fiber*	*Nitrogen-free extract*
Florida	6.00	3.21	18.75	54.00
Texsun	5.50	2.50	10.50	62.00

Davis and Kemmerer (1948) added 4 pounds daily of dried grapefruit peel to an alfalfa-hay ration, and obtained an increase in the amount of milk produced by dairy cows.

Becker *et al.* (1946*b*), in their nutritional experiments with beef and dairy cattle, reported on a feeding project using plain, fresh citrus pulp, pulp mixed with cut Natal grass or cut sugar cane, and citrus press cake. The palatability of citrus-pulp ensilages depended upon that of other feeds to which the cattle were accustomed. Citrus press-cake ensilage was quite palatable. The 2 feed mixtures, pulp with cut Natal grass and pulp with cut sugar cane, were eaten readily, while plain, fresh citrus-pulp ensilage was the least palatable of the 4 diets. The feed flavor was transferred through dairy cows into milk when press-cake ensilage was fed just 2 hours before milking. This flavor was similar to that derived from corn or kaffir ensilage. Such mild flavors were eliminated by regulating the time of feeding.

Kirk *et al.* (1949) attempted to fatten steers by using fresh grapefruit in the feed ration. Fresh quartered grapefruit, 40 to 50 pounds, plus 5 to 7 pounds of grass hay and 3 pounds of cottonseed meal, were fed daily for 120 days. The steers readily ate the fresh grapefruit after the first few days, but its high water content (86.36 per cent) made it impossible for them to eat sufficient nutrients to make gains comparable with those made when fed on dry rations. The average daily gain in weight of steers fed on the fresh grapefruit ration was 1.85 pound, whereas the daily gain of those fed on dried waste plus 2 pounds of ground snapped corn was 2.99 pounds.

Glasscock *et al.* (1950) reported that citrus-seed meal, when fed to growing and fattening beef steers as a part of their protein diet, appeared to have no toxic effect; on the contrary, they thrived on it. However, when the meal was fed to swine, it decreased their rate of gain in weight, even when fed at levels as low as 10 per cent of their total ration.

Seltzer (1950) reported that dried grapefruit waste cost only about 75 per cent as much as rolled barley and other similar carbohydrate concentrates;

also, that many were claiming that dried citrus wastes had a conditioning effect not found in grain feeds.

In dried citrus feed analyzed during the 1950–51 season, Brock and Holleman (1951) determined the following percentage averages, on a dry-weight basis: crude fat, 3.65; protein, 6.87; crude ash, 6.51; and crude fiber, 12.70

Driggers *et al.* (1951) found that citrus-seed meal had to be detoxified before it could be used either as or in a chick feed. They stated that this toxic factor appeared to be limonin—the bitter principle of the seed. Whatever the toxic factor might have been, they found that it could be extracted with diethyl ether, acetone, and 95 per cent ethyl alcohol; the extractions were made successively. After removing the factor, up to 20 per cent of the total diet could consist of the treated meal with good results.

Hendrickson and Kesterson (1951) presented the following percentage values for "a typical feed analysis of dried citrus pulp": crude protein, 6.2; crude fat, 3.5; crude fiber, 13; N-free extract, 63; ash, 4.3; and dry matter, 92. These values were the averages of those given by Neal *et al.* (1935*b*), Pulley and von Loesecke (1940), Arnold *et al.* (1941), Copeland and Shephardson (1944), Kirk *et al.* (1949), and Ralph L. Miller (1949).

In feeding trials using wethers, Ammerman *et al.* (1965) determined the digestibility of protein and the energy production in citrus pulp, citrus meal, and citrus-meal pellets, with particular emphasis on the influence of drying temperatures on the digestibility of protein and energy in citrus pulp. In experiment I, the digestibility of dietary protein in the rations containing any one of the dried citrus-pulp samples varied from 84.1 to 56.6 per cent (table 181). The 3 rations containing dried citrus pulp were equivalent in digestibility, ranging from 84.1 to 77.6 per cent. Those rations which contained either citrus meal or citrus-meal pellets had significantly lower digestibility values than dried citrus pulp, ranging from 70.3 to 56.6 per cent. The energy utilization for the same samples ranged between 82.4 and 64.9 per cent. The energy in rations containing citrus meal or citrus-meal pellets was significantly less utilized than in rations of dried citrus pulp, but the differences in energy utilization between rations were smaller than those between rations in protein utilization.

As a result of the adverse effects of excessive high temperatures on the nutritive value of feeds, experiment II (table 181) was confined to citrus-pulp samples dried at 220, 240 and 260° F. (104, 116, and 127° C.). The digestibility of protein and the utilization of energy in the rations containing citrus pulp dehydrated at 220° F. were significantly greater than in those rations containing pulp dried at 240 and 260°. Protein utilization was significantly higher in experiment I than in experiment II, which could have been attributed to a greater quantity of total protein being supplied by soybean meal. The utilization of energy derived from rations containing approximately

70 per cent citrus pulp was not significantly different between the 2 experiments, indicating that the energy utilized was not greatly affected by the level of dietary protein.

Adding Nitrogen to Citrus Waste. Hart *et al.* (1939) performed extended feeding experiments with urea and ammonium bicarbonate on growing calves, and concluded that the nitrogen of such simple compounds could be utilized as a partial supply of protein nitrogen. They suggested that microorganisms in the ruminant's stomach would convert the simple nitrogen compounds to amino acids, which would then become available to the animal. For further details, see Magruder *et al.* (1953), who investigated the effects of ammoniated industrial by-products in dairy-heifer rations.

TABLE 181

EXPERIMENTS PERFORMED WITH WETHERS TO DETERMINE THE DIGESTIBILITY OF PROTEIN AND THE ENERGY SUPPLIED BY DRIED CITRUS PULP AND MEAL.*

EXPERIMENT I

Digestibility of protein and energy in rations containing dried citrus pulp, citrus meal, or citrus meal pellets.

Major ration ingredients†	Digestion coefficients‡	
	Protein	Energy
Citrus pulp	84.1[a]	82.4[a]
Citrus pulp	81.6[a]	80.6[ab]
Citrus pulp	77.6[a]	80.1[ab]
Citrus meal	70.3[b]	77.2[bc]
Citrus meal pellets (light)	62.3[c]	73.0[c]
Citrus meal pellets (dark)	56.6[c]	64.9[d]

EXPERIMENT II

Digestibility of protein and energy in rations containing citrus pulp dried at different temperatures.

Drying temperatures		Digestion coefficients	
Degrees F.	Degrees C.	Protein	Energy
220	104	62.7[a]	81.0[a]
240	116	50.0[b]	74.1[b]
260	127	50.3[b]	76.9[b]

*From Ammerman *et al.* (1965).

†In Experiment I, citrus pulp, meal, and pellets collectively comprised 72.5 per cent of the total ration. In Experiment II, citrus pulp comprised 70 per cent of the total ration.

‡Means in the same column bearing different superscripts are significantly ($P < 0.05$) different. In Experiment I, each mean represents an average of 4 values. In Experiment II, each mean represents an average of 3 values.

One research worker mentioned briefly a process known as "ammonium pectation" of lemon waste. In this process, ammonia reacted with lemon waste to produce a feed of enhanced protein value for ruminants. It was possible to increase the crude protein of dried lemon pulp from 6 per cent to a total protein equivalent of 12 per cent.

Maurer *et al.* (1951) utilized a method by means of which they could treat citrus waste so that it contained 6 to 40 per cent of crude protein. This was done by simply treating the dehydrated waste with anhydrous ammonia under normal atmospheric pressure and controlled temperature. For each pound (0.454 kilogram) of anhydrous ammonia (fertilizer grade) added to the dried citrus waste, 5.15 pounds of "synthetic" or crude protein were formed. These synthetic proteins were utilized by ruminants (cattle and others) as efficiently as native proteins supplied by a grain-cottonseed ration.

Essential Oils as Commercial Products

THE ESSENTIAL OILS of citrus fruits are commercially important in that they are used as flavorings in both the food and beverage industries. In the natural state, the oils are located in glands or sacs that vary in size from 0.3 to 0.6 millimeters in diameter, and occur at irregular depths in the flavedo or outer peel of the fruit. The fact that the oils are located in specialized receptacles in the outer peel and are not diffused throughout the fruit tissues makes it possible to extract them at various steps in the manufacture of juice products.

Cold-Pressed Oils. The oils are removed from the peel by either the cold-press or distillation methods, but the cold-pressed product is the only one that has detailed specifications in the United States Pharmacopoeia.

The essential oil of grapefruit may be expressed or distilled from the peel of both grapefruit and shaddock. It has been produced commercially in limited volumes in the United States, South Africa, Israel, and Spain. The cold-pressed oil is amber-colored. Owing to the large size of the individual fruits, the yield of oil is much smaller than that obtained from other and smaller citrus fruits. Since oils occur in the flavedo, the amount of oil recovered from the peel is highly correlated with the surface area of the fruit. Consequently, small fruits would yield more oil than large ones, because the former would have greater surface area per unit than the latter.

Nelson and Mottern (1934) noted that grapefruit oil contained approximately 90 per cent D-limonene, and that the terpeneless fraction prepared from it amounted to 2.7 per cent of the total oil content.

According to Hendrickson and Kesterson (1965), cold-pressed grapefruit oils are produced in considerable volume in Florida. They are composed of mixtures of hydrocarbons, oxygenated compounds, and nonvolatile residues. A more detailed discussion of the chemical characteristics of grapefruit oils will be found in the section on essential oils in chapter 3.

The methods for producing cold-pressed citrus oils include such alternatives as: (1) recovery of oil from peel after extraction of the juice, (2) simultaneous extraction of juice and oil emulsion from whole fruit, and (3) recovery of oil from oil-bearing flavedo which has been previously removed from whole fruit by abrasion.

As noted by Braverman (1949), all extraction methods are primarily concerned with ways and means of piercing and rupturing the oil glands, and collecting the oil as quickly as possible to prevent the spongy albedo from absorbing it. The oil-extraction equipment is usually supplied with facilities to keep the surface of the fruit wet with water sprays. These carry away the released oil in the form of an emulsion, prevent loss of oil by spurting, and fill the spongy cells of the flavedo and albedo surrounding the oil glands, thus preventing them from reabsorbing the liberated oil. Finally, the water increases the turgor pressure of the cells of the oil glands by changing the osmotic concentration of the surrounding cells. This results in changes in cell volume caused by the gain or loss of water.

After the oil is expressed from the peel by various procedures, it is flushed from the presses and then removed from the effluent by various degrees of centrifugal separation. After the oil emulsion is put through a revolving reel (a screening device) to remove the larger particles, it is passed through a high-force, disk-type centrifuge, known as a "sludger," which removes additional suspended materials and water. The creamy oil from the sludger is next sent through a centrifuge cycle at 15,000 rpm, which yields a 99 per cent oil discharge. A further refinement is made by centrifuging the oil a third time through a disk-type separator, which yields an oil of 99.9 per cent purity. The refined oil is transferred to tin-lined or stainless-steel drums, and is held in cold storage to precipitate sterols and waxes, which can be separated by decantation. The decanted oil is given a final polishing by centrifuging, after which it is stored in containers that are completely filled with oil, thus resulting in the absence of air.

Methods of Oil Extraction. With the continued increase in labor cost in the United States, the processors of citrus fruits find it important to keep improving the manufacturing procedures that are so essential to the commercial production of their products. With reference to the extraction and recovery of the essential oils from citrus fruits, the research and development programs have been designed primarily for large-scale operations. Cold-pressed citrus oils are produced by methods in which the oil is recovered from the peel after extraction of the juice, and by methods that simultaneously extract the juice and oil emulsion from whole fruit. To express the oils from citrus peel, the glands must be punctured by pressing or rasping.

Hendrickson and Kesterson (1965) investigated the methods of oil extraction used in Florida, and reported that 6 different types of commercial equip-

ment had been used to express citrus-peel oils. These various mechanical devices are described here.

Pipkin-Roll Method of Extraction. This extractor consists of 2 stainless-steel drums (or rolls), turning in opposite directions, which are adjusted to control the pressure on the peel. The pressure is kept strong enough to puncture the oil glands without breaking or rasping the peel. The surfaces of the rolls are grooved at sufficient depths to receive and keep the oil out of contact with the peel, because the albedo has a strong tendency to absorb oil.

Screw-Press Method of Extraction. Some biological materials are of a spongy nature, and may contain large quantities of substances in their tissues in liquid form. Citrus peel is a good illustration of a material in which the oil is incorporated as a liquid in specialized vesicles in the tissue. When such a condition exists, it is possible to make a mechanical separation of the oil from the solid constituents of the peel by the use of a screw press. Tapered screws press the peel against a perforated screen, thereby squeezing out the oil. Water may or may not be used during the operation. Presses will vary in design; some are mounted horizontally, while others are mounted vertically.

Fraser-Brace Extractor. In this method, whole fruits are passed over carborundum rolls which rasp their surfaces during rotation. Water spray is run over the fruit and the rasped surface to wash away the oil and grated peel. The oil–water emulsion and the abraded raspings are conveyed over a screen to remove the suspended solids. The emulsion is then transferred to settling vessels to allow time for separation.

FMC Rotary Juice Extractor. This extractor was once widely used by citrus processors, and it was successful in the extraction of both juice and peel oil from whole fruit without the 2 phases coming in contact with each other. This equipment has been withdrawn from the market and replaced by the FMC In-Line Extractor, which is more efficient in keeping the juice and oil phases separated during the process.

FMC In-Line Extractor. Although this machine is especially adapted to the internal extraction of juice from whole fruit, an oil-cup assembly can be inserted in the system to extract oil from the peel and form an oil–water emulsion at the same time that juice is extracted. In this method, the fruit is fed singly into stationary extraction cups, where it is automatically centered and positioned for extraction. As the upper half of the cup is lowered, the metal fingers of the 2 halves intermesh, thus applying equal pressure to all areas of the fruit surface. In the bottom of the lower cup, a stainless-steel cutter tube is connected to the finishing tube and manifold. The cutter tube cuts a circular plug through the peel in the bottom of the fruit and, as pressure is applied by the intermeshing cups, the internal pulp and juice of the fruit are squeezed through the opening into the finishing tube, where a rising orifice tube begins the prefinishing operation. The residual compressed pulp, section membranes, seeds, and dry plugs are ejected from the lower end of the orifice tube as the juice and juice cells flow through the perforated walls of the prefinisher tube into an enclosed juice manifold. This method extracts juice from whole fruit without contacting the outer peel, and instantly separates the juice from the undesirable pulp solids. The distinguishing characteristic of this juice extractor is the strainer tube, which serves as a prefinisher. With this tube, the section membranes, core, and seeds are firmly pressed and eliminated immediately from the juice, without the necessity of removing them by standard-type finishers. The strainer tube makes it possible to obtain maximum juice yields without incorporating an excessive amount of pectic substances and pectic enzymes (Gerwe, 1954).

AMC Scarifier. Machines for scarifying and rasping whole fruit for the production of peel oil have been developed to various degrees of sophistication in the citrus-growing areas of Mediterranean countries. The *écuelle,* a metal funnel covered on the inside with short spikes and used in obtaining essential oil from citrus fruits, can be regarded as the prototype of all machines used for rasping whole fruit (Braverman, 1949). The American Machinery Corporation has designed a scarifier for continuous operation which is inserted in the conveyor line to the juice extractor. This machine consists chiefly of a hollow stainless-steel cylindrical drum, with its interior surface made rough by piercing it from the outside with a square punch which produces sharp edges on the extrusions that project at right angles with the interior wall. These sharp edges serve the purpose of puncturing the oil glands of the peel. As the drum rotates, a continuous water spray is delivered inside the drum to maintain a saturated atmosphere. The fruit enters the lower end of the drum, rotates, and rubs against the sharp edges, and is subsequently forced out of the drum at the elevated end. The oil–water emulsion and the flavedo scrapings are washed through the holes punched in the drum, and the emulsion is collected in a stainless-steel pan beneath it.

Distilled Oils. Oil that remains in the peel and effluent after pressing and separation is removed by distillation. The distilled oils of oranges and grapefruit are obtained as a result of processing these fruits for juice. The liquid discharged from a cold-pressed oil centrifuge usually contains approximately 0.1 per cent oil, which may be recovered by steam distillation, and the condensed oil and water vapors are collected in drums from which the oil is decanted from the top of the water layer. The residual oil that remains in the peel after subjecting it to cold-pressed procedures may be recovered by steam distillation. These steam-distilled citrus oils are inferior to cold-pressed oils, both in flavor and keeping characteristics. In other words, there is a marked difference in the chemical and physical properties of the two.

"Stripper oil" is a distilled oil obtained as a by-product from the manufacture of citrus molasses. The term applies specifically to the peel oil that is stripped or recovered by distillation from citrus press liquor. In Florida, the press liquor is usually derived from a mixture of citrus fruit varieties; consequently, the stripper oil recovered represents a mixture of steam-distilled oils. It contains 95 per cent or more of D-limonene. This oil has been used in certain types of plastics and in the preparation of "isoprene."

Distilled oil can also be obtained by de-oiling citrus juices that contain higher quantities of oil than is necessary. The juice enters a heat exchanger maintained at 125° F. (52° C.) and under a 26-inch (66-centimeter) vacuum. The vapors containing the oil are condensed and passed into a decanter, where the oil separates and is drawn off as the top layer. This method removes approximately 80 per cent of the oil present in the juice. To control the amount of peel oil in the juice, de-oiling becomes necessary, and this may be accomplished by vacuum distillation. Distilled oils obtained in this way resemble those obtained by steam distillation at atmospheric pressure.

Pectin

PECTIN OCCURS in most fruits in varying amounts. The quantity of pectin present is usually small, being chemically associated with other carbohydrate substances in the fruit tissues. This makes the production of high-quality pectin impracticable in most cases. In citrus fruits, however, pectin occurs chiefly as protopectin in the white albedo portion of the peel, in the cell walls of the juice vesicles, and in the fibrovascular bundles. The albedo contains approximately one-third of its dry weight as pectic substances and one-third as cellulosic material, while the remainder is composed of flavonoids, polysaccharides, and monosaccharides such as glucose and fructose. The amounts of pectin that exist in these tissues show significant variations between different varieties of citrus fruit as well as between different degrees of maturity. The most suitable varieties for producing commercial pectic substances are lemons, grapefruit, pummelos, and oranges. Grapefruit peel is very rich in pectic substances, and is one of the best sources of commercial pectin. According to Rygg and Harvey (1938), the albedo was found to contain more soluble pectic constituents than the flavedo, but the total pectic materials changed very little during growth of the grapefruit. The commercial producer of pectin can utilize only a limited amount of the peel residue that is available for conversion to by-products. For several decades, the Sunkist organization in California has produced large tonnages of orange and lemon pectic materials for commercial purposes.

The Commercial Production of Pectin. The general outline for the production of pectin from citrus peel includes the preparation of the peel for extraction: first, by removing the oil and subsequently macerating or mincing to facilitate washing and extraction; and secondly, by leaching with cold water to remove soluble sugars and glycosides. The next steps include the hydrolysis of protopectin in the peel to soluble pectin, filtration of the pectin extract, and precipitation of the soluble pectin from the extract, followed by a system of purification and drying of the final product. This outline serves as a basis for summarizing more fully the methods for producing citrus pectin from both lemons and oranges.

Citrus peel is usually the raw material from which pectin is produced. The macerated peel, from which the oil and juice sacs have been removed, is flumed to the pectin-extraction equipment. This operation not only conveys the material, but also permits the flume water to leach the citric acid and other soluble constituents from the peel. The presence of citric acid in the peel interferes with the complete precipitation of pectin from the clarified liquors in the older procedure, where the "alum process" is used to form a colloidal complex of aluminum ion and pectin. The presence of citrates and tartrates inhibits the formation of pectin (Hull *et al.,* 1953).

After removing the flume water in a rotating reel, the shredded peel is conveyed to extraction tanks equipped with sweep agitators, where a measured amount of water is added to each tank of slurry. The contents are then heated to the boiling point with saturated steam introduced directly through perforated pipes. After adding concentrated hydrochloric acid to reduce the pH below 3.0, the mixture is cooked for 30 to 40 minutes, the time during which insoluble protopectin is hydrolyzed to soluble pectin. The pectin-containing liquor is separated from the pulp mass by a drum filter. The liquor is then pumped through plate and frame filters, using diatomaceous earth as a filter aid. The clarified liquor from the frame filters is concentrated in upflow vertical-tube double-effect evaporators, with steam on the first effect, using vacuums at 12 and 26 inches (30 and 66 cm). The pectin liquor is concentrated in the vacuum pans from a 1 per cent to a 3 per cent concentration. This solution is sprayed into a tank, where precipitation occurs at a concentration of 1.5 part of isopropyl alcohol to 1 part of pectin solution. The precipitated pectin is removed from the solvent by means of a hydraulic press, roller press, or drain-screw conveyor. The crude pectin is then sent through several washings in 50 to 70 per cent alcohol–water mixtures, and is finally dehydrated in 85 per cent alcohol. The washed pectin is further pressed to remove solvent and water up to 50 per cent of the amount present. After drying the pectin in a circulating air drier or on a heated drain to a moisture content of 6 to 10 per cent, it is finally pulverized and sifted through a 60-mesh screen.

In the so-called "alum process," the extraction of pectin from the protopectin of the albedo is accomplished by the use of dilute sulfurous acid. This acid is volatile, and the excess can be removed from the pectin liquor by aeration, thus eliminating the salts that would be formed by neutralization of the acid. Sulfurous acid produces a sufficiently low pH for the extraction and, after aeration, the pH increases without the addition of buffers or alkali. In this process, solutions of aluminum chloride and sodium carbonate are added to the clarified liquor to adjust the pH for precipitation of green pectin. The optimum precipitation occurs at approximately pH 4.0, varying between pH 3.8 and 4.2. The aluminum chloride hydrolyzes in the pectin liquor to a basic salt that stays in solution until the pH reaches about 3.5, when aluminum hydroxide is formed. The colloidal aluminum hydroxide forms a precipitate with the pectin in the form of a yellowish-green curd. This complex precipitate (pectin and aluminum hydroxide) requires several complete washings with acidulated alcohol to dissolve the firmly bound aluminum from the insoluble pectic materials (Joseph and Havighorst, 1952).

Types of Pectin. Since pectin is considered to be a long chain composed of polygalacturonic acid units with carboxyl groups partially esterified with methoxyl groups, it follows that the important physical and chemical proper-

ties of pectin are highly associated with its molecular size, degree of esterification, and the galacturonic acid content in relation to other accompanying carbohydrates.

The most extensive use of pectic materials is dependent upon their ability to form gels in systems, either of the usual high-sugar type or of the low-solids type. The practical aspects of gel formation with both high- and low-ester pectinates are of extreme importance to commercial fruit preservers and food processors.

Regular commercial pectins normally produced by the process just described are the so-called rapid-set pectins, which gel very quickly, even at temperatures near the boiling point, provided the pH is between 2.0 and 3.5 and sucrose is present to approximately 65 per cent of the total weight. This type of pectin is desirable in the manufacture of jams, marmalades, and preserves because it causes gels to set rapidly where it is important to have thickening and gelation occur before the fruit and pulp particles rise to the top of the container. In these high-solid gels, it should be noted that the rate of gelation is also a function of several variables, such as sugar concentration, amount and type of pectin, pH, and temperature. The commercial rapid-set pectins have a high degree of polymerization, and the setting time is greatly influenced by the degree of esterification, as well as by the pH of the gel mixture.

The slow-set pectins are produced by changing the normal commercial procedure for production of pectin. The pectin extract obtained by the standard procedure is allowed to react in a hydrochloric acid–alcohol mixture for 10 to 12 hours at 35° C. (95° F.). Under these conditions, the temperature and time determine how slowly the finished product will set. It has been suggested that this treatment probably produces a slight decrease in esterification, accompanied by a slight reduction in molecular weight or chain length, which results in slow-setting properties of the pectin produced. The advantage of using slow-set pectin in jellies is that the acid required to set the jelly can be added all at one time to the cooked batch without the fear that the jelly will begin to set before it is poured into containers, as would generally be the case if rapid-set pectin were used.

Low-methoxyl pectins are different from regular pectins or high-methoxyl pectinates in that they do not require sugar for gel formation, and are not so sensitive to pH as are the regular pectins. This means that the hydrogen ion concentration of gels made with low-methoxyl pectin (Pectin LM, commercially) is not so important as it is with regular high-sugar pectin gels. The degree of esterification of low-methoxyl pectin has its upper limit at 7 per cent methoxyl, but the low-ester values of certain commercial pectins usually vary in methoxyl content from 3.5 to 5 per cent by weight. If all the methoxyl groups are removed from the galacturonic acid chain, the pectin becomes pec-

tic acid or a pectate, which subsequently serves no further purpose as a gel-former in the usual type of jelly or preserve. In contrast to complete demethylation, pectin which has had 50 per cent or more of its ester groups replaced by carboxyl, as illustrated by low-methoxyl pectin, becomes highly sensitive to polyvalent cations, and ionic bonding or cross-linking through primary valences are reasonable possibilities.

In the low-methoxyl pectins, the presence of polyvalent cations binds adjacent chains together, forming a gel framework through ionic bonding. The monovalent cations, such as sodium and potassium, react easily with carboxyl groups instead of bonding to nearby molecular chains. The presence of sodium or potassium prevents calcium from forming a gel structure, because interference with cross-linkages has occurred, and additional time is needed for the calcium to form a random gel structure.

Low-methoxyl pectins are prepared by altering the regular process used for the commercial production of pectic substances. At a selected stage in the production process, the pectin can be demethylated with hot concentrated acid, cold alkali, or the enzyme pectinesterase. Low-methoxyl pectin or pectic acid, depending on the degree of de-esterification, will precipitate in acid solution, and they can also be precipitated from an alkaline solution by acidifying and adding traces of calcium or other polyvalent cations. The low-methoxyl pectin precipitate can be purified by treating it with several washings of acidulated alcohol, and finally with acidulated 70 per cent alcohol, which has proved to be sufficient to remove all traces of cations.

Regular high-methoxyl pectin can be partially demethylated with acids, fixed alkalies or dilute aqueous ammonia systems, enzymes alone or in combination with alkalies, or ammonia in alcoholic or concentrated aqueous ammonia systems (Joseph, 1955). Although low-ester pectinate can be produced by each of the reagents, the product obtained from the ammonia systems has resulted in substituting acid amide groups for some of the ester groups. This low-ester pectinic acid amide is the low-methoxyl pectin (Pectin LM) that has been produced commercially for many years.

Pritchett (1968) reported that an improved type of low-methoxyl pectin had been made available under the registered trademark of Hi-Poly Pectin (Research and Development Department, Sunkist Growers, Inc., Los Angeles, California). This new pectin has the characteristics of forming gels of excellent resilience, resistance to syneresis, and exceptionally fine texture, either with or without sugar. With this particular type of pectin, the pH is not of primary importance as it relates to the gel-forming system. Excellent low-solid gels can be produced at pH 3.0 to 3.5 with a calcium ion concentration of 45 milligrams per gram of pectin. At pH 4.0, the maximum gel conditions are partially related to the buffer system involved. This low-methoxyl pectin can make good gels with milk in the range of pH 5.0 to 6.0 with the milk sup-

plying the calcium, the polyvalent cation requirements. Evidence seems to indicate that these high-polymer pectins with intermediate ester contents can produce regular sugar–pectin–acid gels similar to those obtained with high-methoxyl pectin. The exceptional physical and chemical properties of this gel-forming pectin are related to the large size of the galacturonic acid molecules, the relatively low methoxyl content, and the necessary presence of calcium or other cations to form gels by cross-linkages with the pectin molecules. This product has a wide variety of uses, such as in dietetic jams and jellies where low concentrations of sugar or nonsugar products are used as sweeteners, dessert and salad gels, milk puddings, high-solid candies, baby-food gels, gelled fruit cocktails, and many other food combinations.

Standardization of Commercial Pectins. Pectic materials that are used in foods are usually standardized on their abilities to form fruit jellies. Consequently, the criterion of performance is the "jelly grade," which is defined by the ratio of the weight of sugar to the weight of pectin in a normally firm jelly with 65 per cent soluble solids. Other characteristics, such as setting time, setting temperature, pH, particle size, odor, flavor solubility, ash, and moisture, are some of the important factors governing the formation of sugar–acid–pectin jellies, but it appears that jelly firmness, based on the results of extended experimentation, is the most important single factor for standardization of commercial pectins. Low-methoxyl pectins of the older type were usually standardized so that 1 gram of pectin would require 25 milligrams of calcium to form a standard gel with a 30 per cent sugar solution at a fixed pH and temperature.

Calcium Sensitivity in Relation to Pectin Gel Characteristics. Researchers who have had considerable technical experience in the commercial production of pectin from freeze-damaged citrus fruit have often observed the extreme sensitivity of pectic substances to divalent cations and alkaline earths. The freezing of citrus fruits which usually occurs during the winter months of the growing season has made some physical alterations in the pectic constituents, yet the changes are not revealed by the present analytical techniques. Changes other than freeze damage may also occur in the fruit during its growth and maturation as a result of environmental factors that may accelerate the calcium sensitivity of the isolated pectic substances. Experience is the source of the suggestion that pectic substances react to varying degrees with alkaline earths, but data are not available to show how the reactivity alters with cultural, weather, and other growing conditions of the fruit. However, it is fairly well known that calcium sensitivity in relation to characteristics of pectin gels is recognized as occurring in the juice vesicles of Valencia oranges and varieties of grapefruit that have undergone the abnormal development commonly referred to as "granulation." Granulation begins by an increase in volume of the juice vesicles with hydration of the cell contents,

followed by gel formation of the pectic substances. Gelation is a process through which all juice vesicles pass before reaching the stage where they begin to disintegrate and desiccate. The experimental evidence presented indicates that the gelation is the result of the combination of the divalent calcium ion with the pectic materials to form hydrated calcium pectate. The amounts of calcium and magnesium are especially large, there being an average of only 46.9 per cent as much calcium and 59.1 per cent as much magnesium in the healthy juice sacs as in the granulated ones. Furthermore, the granulated vesicles contain 43.7 per cent of total calcium as water-insoluble calcium, whereas the average for healthy vesicles is 34 per cent. As the concentration of sugars and organic acids decreases, the pH of the juice increases, with an increase in the mineral constituents available for salt formation. With diminution of these materials, the gelation process in the affected vesicles does not depend on the high sugar content or low pH, but probably depends on the presence of divalent cations, such as calcium, to react with any available amount of demethoxylated pectic materials (Sinclair and Jolliffe, 1961).

As usually understood, gel formation requires 4 constituents: sugar, acid, pectin, and water in a definite relationship to each other. In this particular system, pectin is the gelling agent, sugar and acid are the agents that bring about this physical transformation, and water acts as a solvent for the other 3 constituents. However, in granulated juice vesicles, these 4 ingredients are not present in the right proportions for this particular type of gel. The concentrations of sugar and acids in the affected vesicles are insufficient, but enough pectic materials are present with free carboxyl groups to form gels in the presence of divalent cations. Both the pectic substances and their respective methoxyl contents increase with the severity of granulation. This increase in the percentage of methylation of the pectic material would generally be associated with greater pectin solubility; however, sufficient cations are available to overcome this effect and cause gelation.

Pectate Pulp. Pectate pulp is not a highly refined pectin, but it is a leached peel product which does contain principally higher-polymer carbohydrate materials such as low-ester pectins, pectic acids, hemicelluloses, crude celluloses, and other cell-wall constituents of high molecular weights. Pectate pulp is made by treating citrus peel with soda ash, and the resultant product is washed, pressed, dried, and ground. When the citrus pulp is treated with an alkali, the pectin is converted to a pectate, and the natural calcium of the pulp reacts with the soluble section to produce water-insoluble calcium pectate in the cellulose matrix of the pulp. This material is dispersible in hot water in the presence of suitable amounts of sodium phosphate. Dispersed systems of pectate pulp can be made to flocculate over a wide range of acid and alkaline pH, thus serving the purpose of removing or recovering suspended particles (Hull *et al.*, 1953).

Pectic acid pulp is an unrefined peel product which differs from pectate pulp in that dispersions in the aqueous phase can be easily obtained by adding soda ash or ammonia. When the citrus pulp is treated with acidulated alcohol at low pH values, there results a pulp with either low or high viscosity, depending upon the degree of depolymerization of the cellulose–pectin complex.

Pectin pomace, a product made from citrus-peel residue, was commercially produced in Florida in 1944, and was used by the jam, marmalade, and jelly industries (Hendrickson and Kesterson, 1965). This product was made by a method developed by Pulley *et al.* (1944), using peel residues from extracted grapefruit. The process consisted essentially in extracting the shredded peel residue with boiling water to inactivate the enzymes and remove the soluble carbohydrates and other materials from the peel. The minced material was given 2 additional extractions with cold water, and the second extract often contained aluminum sulfate, a polyvalent cation, which dehydrated the water-insoluble residue, thus facilitating the removal of water by pressure. The leached peel residue (sold as "Pectin Pomace") was finally dried to 4 to 8 per cent moisture and ground to pass a 20-mesh screen.

Commercial Uses of Pectic Substances. Large tonnages of pectin for food, pharmaceutical, and industrial uses are made from citrus peel after the removal of oil. The principal uses of the pectic substances are in the commercial and home production of jams and jellies. The best-known applications are based on their colloidal properties in the making of jellies, candies, and other food products. Pectins are used to stabilize emulsions in cold creams, other cosmetics, and soaps. They are used as thickeners and stabilizers in milk products. The emulsifying properties of pectin have led to its use in mayonnaise, salad dressings, and flavoring oil emulsions. Some practical applications of low-methoxyl pectins have already been mentioned.

Preserved Products

MARMALADE PRODUCTION from citrus peel and juice is not only another way to preserve the fruit for future use, but it is a way to use citrus peel as a by-product for human consumption. Citrus marmalades that are produced in the United States are usually clear jellies in which sliced peel is suspended. The British marmalades, similar to home-prepared products, are cloudy as a result of suspended colloidal materials. This opaque type is prepared by cooking the edible portion of the fruit, both juice and tissues, in a small volume of water. Then, after removing the seeds and locular membranes, the acidity of this base is regulated to a pH of 3.0 to 3.4 with citric acid. The next step is to add previously tenderized peel and adequate amounts of sugar, and cook to the required end-point (Braverman, 1949).

The jelly base for a clear marmalade can be produced from reamed or processed juice. The juice is screened through a fine finisher and then strained through a muslin cloth, which process will prepare it for a clear marmalade. After adjusting the pH (3.0 to 3.4) with citric acid, the required amount of juice is mixed with previously tenderized peel, and the calculated amount of sugar is added in increments during the boiling process. Prolonged boiling of the marmalade components at relatively high hydrogen ion concentrations hydrolyzes the sucrose to invert sugar, the presence of which prevents the crystallization of the sucrose in marmalade during storage. The pectin should be added as late as possible during the cooking process, in order to avoid the hydrolysis of pectin to pectinic acids or to lower degradation products which have no gelling capacities.

Bitter-orange marmalade base will sometimes gel in bulk containers when stored in warehouses at ambient storage temperatures. Rouse and Atkins (1957) reported that no gelation or only semigelation occurred in base samples adjusted to pH 4.0; and in those samples treated with Pectinol M, either at original or adjusted pH 4.0 values, no gelation occurred in the samples stored for 24 weeks at 10, 40, and 90° F. (–12, 4.4, and 32° C.).

Sedky *et al.* (1942) noted that when marmalade was prepared by using orange concentrate, it was possible to reduce the time required for production, thus resulting in a greater protection of the vitamin C content and flavor of the finished product. The amount of ascorbic acid in the marmalade could be controlled by adjusting the quantity of juice used. It was concluded that marmalade prepared by this procedure retained 80 per cent of the ascorbic acid content of the juice in the finished marmalade. The use of separately cooked peel would protect the juice from the detrimental effects of prolonged cooking, and thus would retain the color, flavor, and vitamin C content of the juice.

According to federal standards, citrus-fruit marmalade should contain not less than 68 per cent soluble solids. Marmalade produced from sweet oranges should contain not less than 30 parts by weight of fruit to 70 parts by weight of sugar, while the bitter-orange marmalade should be composed of not less than 25 parts of fruit to 75 parts of sugar. The peel content of citrus marmalade is not specified by grade standards, but the amount present must be equally distributed, uniform in size, and in substantial but not excessive amounts.

In the preparation of peel for marmalade production, it is important that it should be cooked in 2 changes of water until tender. This can be done before or after slicing. If the sliced or chopped peel is not completely tenderized before boiling with sugar, it will remain hard and tough in the final product. In addition to tenderizing the peel, the cooking process can also reduce bitterness in grapefruit peel by using 2 or 3 changes of water.

The types of marmalade that appear in United States markets are produced chiefly from the sour or bitter orange and the sweet orange. Infrequently, a product is prepared from a blend of orange and grapefruit. A highly flavored and nutritious marmalade can be prepared from grapefruit, and limited amounts of this product have sometimes appeared in the markets.

Citrus Bland Syrup. Hendrickson and Kesterson (1965) stated that a bland syrup, used to sweeten single-strength citrus juices and canned grapefruit segments, could be made from press liquor, and that bland syrups could be prepared from citrus and other fruit juices. According to Joslyn (1954), "syrup" is a term applied to fruit juices that have been concentrated by removal of water by evaporation or freezing procedures. In the beverage industry, the term is used to designate fruit juices to which sugar has been added or in which the free acids have been neutralized or removed from the juice by ion-exchange resins.

The production of citrus bland syrup is based on the principle of using ion-exchange resins (both cation and anion) that have the capacity of removing, by exchange absorption, the citric acid, minerals, and other impurities present in citrus juices. These syrups have a quality superior to those that have had their free carboxyl groups neutralized by alkali. In addition to ion-exchange treatment, the press liquor is clarified and decolorized, and the dilute pure sugar solution thus obtained is concentrated by conventional methods to the desired Brix, usually 75 to 85°. This syrup is used as a substitute for granulated cane or beet sugar. At the time Hendrickson and Kesterson (1965) reported, the citrus industry of Florida was purchasing approximately 10 million pounds of sugar each year to sweeten single-strength juices and canned grapefruit sections.

The use of syrup from citrus liquors shows an economic advantage over the purchase of raw sugar. Also, liquid sugar solutions are easier to handle.

Sweetened fruit juices can be prepared from citrus-juice concentrates with various mixtures of pure sucrose or sugar syrups, in which various amounts of flavoring ingredients, such as essential oils, citric acid, and coloring substances, are added to compensate for the loss of flavor owing to dilution of the fruit solids. It thus follows that fruit-juice syrups, especially those prepared from a given citrus-fruit juice or blends of different citrus juices, can be used as beverage bases.

Although grapefruit juice has an excellent flavor, the amounts used in beverages have never equaled the quantities of juice used from other citrus species—oranges, lemons, and limes. Grapefruit base is used in variable quantities by the dairy industry, and the beverage produced contains approximately 15 per cent juice. Certain amounts of grapefruit base are used in carbonated drinks as mixers. When the juice is to be used as a beverage base, it is not concentrated over a maximum of 60° Brix, because at higher

levels grapefruit concentrate appears to darken in direct proportion to the degree of concentration under a given storage environment.

Brined and Candied Peel. Raw peel is usually taken directly from the burring room to the brined peel plant, where it is placed in large wooden tanks for fermentation in salt brine. Before fermentation is initiated, the pulp and rag are removed from the peel, which is then processed into cups, strips, or diced form. The product is packed in barrels with salt brine added for shipment to customers. Brined peel is used principally by suppliers to the bakery and confectionary trade, and consequently the volume produced in the United States is limited chiefly to these 2 economic outlets.

The brining of citrus peel (grapefruit, oranges, and lemons) has long been an established practice in the Mediterranean area. In Italy, lemon peel is the raw material that is usually placed in brine. The peel remains in the brine for 4 or 5 days, after which the halves are packed in barrels, one within another, in layers, and stored for 2 months completely immersed in a 10 per cent salt solution. During the storage period, fresh brine is added when needed to maintain constant volume and to keep the peel covered. The degree of curing is evaluated by the translucent appearance of the peel without exhibiting a mealy or mushy condition. This means that the cured or pickled peel is a firm but tender rind which has become transparent while excessive bitterness has disappeared (USDA, 1956).

Either fresh or brined peel can serve as raw material for producing candied citrus products. Citrus peels are usually brined for preservation until they can be converted later into candied products. Fresh citrus peel can be utilized for candying purposes by covering the material completely with water and boiling for stated periods (say 1 hour). After boiling, the water is poured off, fresh water is added, and the boiling is continued until the peel is tender. During the cooking process, several changes of water are required to reduce the bitterness and some of the peel oil. Grapefruit peel has a decidedly bitter flavor, and several extractions with water are required to reduce the bitterness to an acceptable concentration. However, a certain amount of bitterness should remain, for it is a distinct characteristic of this fruit. The tenderized peel is drained, and then covered with varying and increasing concentrations of sucrose solution—up to 50 per cent sucrose syrup containing 0.05 per cent citric acid. The mixture of peel and syrup is boiled slowly for 1 hour and then allowed to stand for about 20 hours, after which it is again brought to a boil at 219° F. (104° C.) and drained while still hot. The drained peel is then dried, and is sprinkled with either powdered sugar or cornstarch. After further drying in air, the candied product should be stored in air-tight containers. Before candying, the brined peels are washed thoroughly in running water to remove the salt. Otherwise, the procedures used for candying brined peel are the same as those used for fresh peel.

Fermentation of Juices and Waste Liquors

A CLARIFIED CITRUS WINE was developed by Cruess (1914) in the course of studies on the utilization of waste oranges. He fermented clear orange juice with pure yeast, and obtained a wine that filtered brilliantly clear, and contained 4.5 per cent alcohol and 1.5 per cent total acid. Its flavor was superior to that of the product made by natural fermentation. It was turned into a sparkling wine with a pleasing taste after a second fermentation in bottles in the presence of additional amounts of sucrose.

Alcoholic beverages can be made by fermenting fruit juices, provided sufficient carbohydrates are present to serve as source material for conversion to alcohol. Oranges, grapefruit, and tangerines are the types of citrus that are most suitable for wine-making, even though the soluble-sugar contents of their juices are not sufficiently great to produce high-quality wines. Although these fruits have a somewhat high acid content, the exorbitant amounts that exist in lemons and limes, coupled with the presence of low concentrations of fermentable sugars, make them unlikely to serve as starting materials for the manufacture of wines.

Alcohol that is obtained from the fermented liquid by distillation is brought to the market under different names, according to the proportion of alcohol and the nature of the by-products present. According to Von Loesecke *et al.* (1936), orange and grapefruit wines can be fortified by adding orange and grapefruit spirits to them to increase their alcoholic contents from 18 to 22 per cent by volume. Whisky or brandy contains 30 to 60 per cent alcohol, and higher concentrations of alcohol for domestic and industrial uses contain from 70 to 90 per cent. Aqueous solutions of alcohol higher than 90 per cent are usually designated and labeled as "alcohol."

As a result of low alcohol content, citrus wines have poor keeping qualities and consequently develop off-flavors in storage. In order to increase the alcohol content of citrus wines, the available possibilities are to increase either the sugar content of the juice before fermentation or to add citrus brandy or alcohol to the finished product. The sugar content can be increased indirectly by concentrating the juice or by adding concentrate to the juice before fermentation.

Von Loesecke *et al.* (1936), using orange and grapefruit juices as experimental materials, determined the correlation between the total soluble solids (Brix) in the juice before fermentation and its alcohol content after fermentation. It can be observed from figure 179 that owing to the lack of dispersion of the experimental points from a straight line, there existed a significantly high relationship between these 2 variables. This strongly indicated that the alcoholic content of the finished wine was a linear function of the quantity of sugar present in the juice during fermentation. These researchers observed

that 55 per cent of the sugars present in the juice became converted to alcohol, provided the fermentation was completed. Consequently, the alcohol yield of a juice was the product of 0.55 multiplied by its Brix reading. This degree of conversion was obtained from experiments using the authors' yeast culture (4097).

Additional data were obtained on the yield of alcohol when various amounts of sucrose were added to orange and grapefruit juices before fermentation. An inspection of figure 180 will show that the relationship between these 2 variables was nearly linear as the production of alcohol increased up to 11 per cent (by volume). Beyond this concentration, the line became curvi-

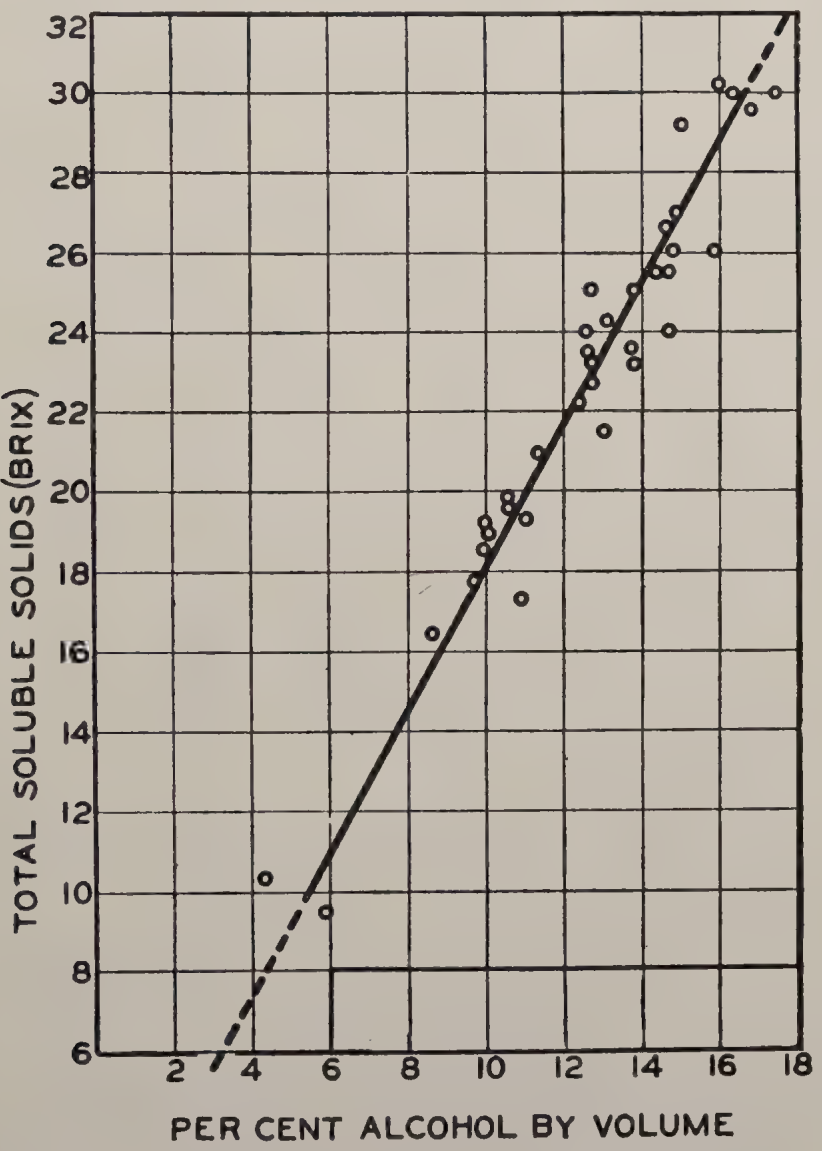

Fig. 179. Correlation between Brix of juice before fermentation and alcohol in juice after fermentation. (From Von Loesecke *et al.*, 1936.)

linear, and between 15 and 16 per cent alcohol, the higher concentrations of sucrose produced no further increase in it. When the alcohol reached a certain concentration during fermentation, the yeast cells were killed or inactivated. Wines were obtained from orange and grapefruit juices with alcoholic contents of 17 per cent (by volume) under these experimental conditions. To produce table wines from the citrus fruits employed, the total soluble solids

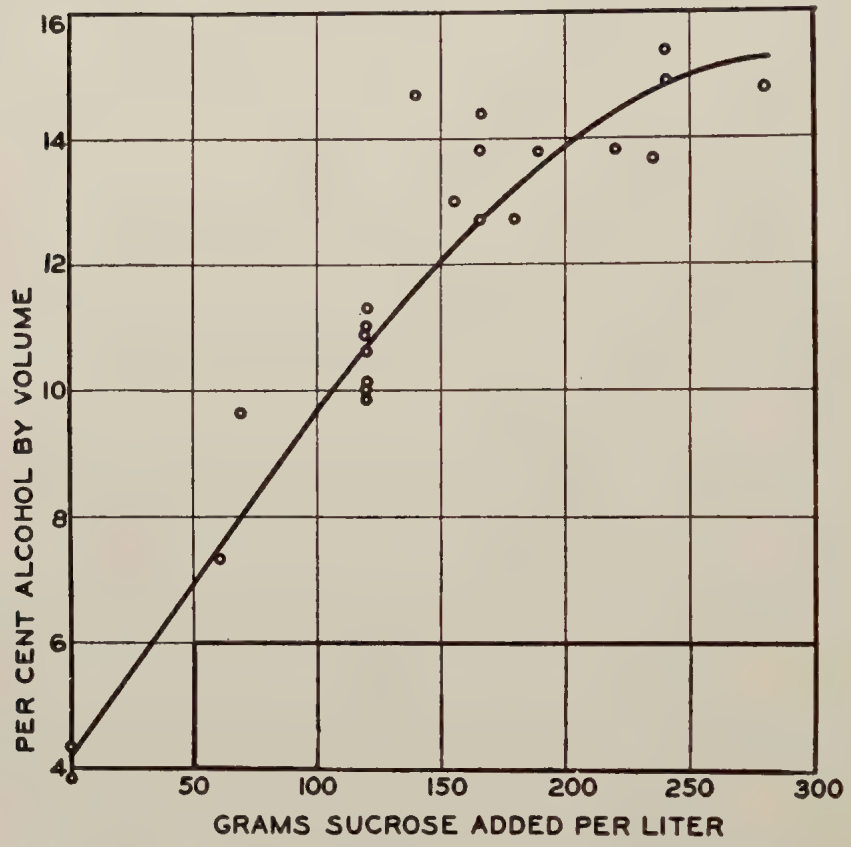

Fig. 180. Yields of alcohol obtained by adding different amounts of sucrose to orange and grapefruit juices. (From Von Loesecke *et al.*, 1936.)

(or Brix) of the juice had to be increased to 24 per cent by adding sugar, which upon fermentation produced wines with approximately 13 per cent alcohol (by volume).

The rate of formation of the products of fermentation is dependent not only upon the concentrations of the reacting substances, but also upon the temperature of the reaction medium. The temperature factor can be illustrated by plotting the alcohol produced by fermenting sweetened orange juice (24° Brix) at different temperatures (figure 181). Fermentation of citrus juices at temperatures above 25° C. (77° F.) produced wines with alcoholic contents that continually decreased with increases in temperature. Citrus wines that were fermented at higher temperatures had not only lower alcohol

contents, but darker color. Von Loesecke *et al.* (1936) emphasized the fact that citrus juices could not be fermented at high temperatures because the presence of acetic acid organisms produced a substance with a distinct aroma of ethyl acetate. They found that the desirable temperature for fermenting citrus juices for wines was about 16° C. (61° F.), and for brandies it was from 24 to 27° C. (75 to 81° F.). As the temperature increased to 35° C. (95° F.) or

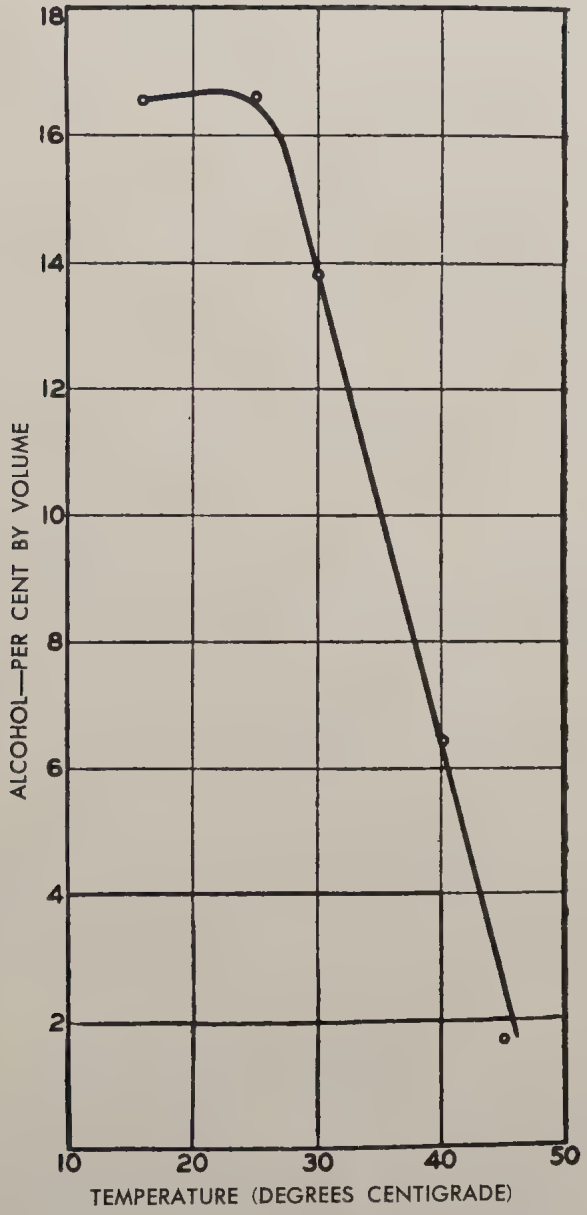

Fig. 181. Yields of alcohol obtained by fermenting sweetened orange juice (24° Brix) at different temperatures. Conversions from C. to F.: 10—50; 20—68; 30—86; 40—104; 50—122. (From Von Loesecke *et al.*, 1936.)

higher during the active period of fermentation, the must or fermenting juice had to be forced through cooling coils to reduce the temperature in order to prevent detrimental effects to the yeast cells, and thus prevent the production of wines with low alcoholic contents.

Citrus juices that are to be used in fermentation should be as free as possible of peel oil, because the essential oils, when present in the juice in sufficient amounts, not only may retard fermentation, but may totally inhibit the conversion of sugar to alcohol. It is also important to centrifuge citrus juices before fermentation. By centrifuging, the pulp is separated from the juice, and the essential oils present in the juice are eliminated.

Since the bitter principle of grapefruit juice was found to be attributable to the glucoside naringin, Von Loesecke *et al.* (1936) reported that fermented grapefruit juice was more bitter than unfermented juice because the sugar content of the unfermented type tended to mask the bitter flavor of naringin. To reduce the unpalatable bitterness of grapefruit wines, these scientists recommended the filtering of the juice clear, before fermentation, by means of a filtering aid and a plate-and-frame press, or by treating the fermented juice with activated charcoal, which reduced the bitterness better than filtration.

Citrus Vinegar. In the production of vinegar, two biochemical mechanisms are involved. First, sugars that are present in the substrate are fermented to ethyl alcohol by the action of yeast, and secondly, the alcohol is then oxidized to acetic acid by acetic acid bacteria. In general, any fruit juice that contains a sufficient quantity of soluble sugars can be converted into vinegar which will contain more than the legal limit of 4 grams of acetic acid per 100 milliliters. Vinegar can be made from citrus juices by 3 methods: a slow or so-called roller process, a rapid-generator process, and the submerged fermentation process.

The rapid-generator process for the production of vinegar is a good illustration of how it is prepared from fermented juices. A tall wooden vat is fitted with 2 perforated disks, located about 4 feet (122 centimeters) from the top and bottom to allow a free flow of air. The space between the disks is filled with beech-wood shavings, or other types of porous materials, such as pomace and corncobs, to obtain efficient and continuous contact between the organisms and the air. The beech shavings serve on the one hand to present a large surface of liquid to the oxidizing action of the air, and on the other to provide a suitable medium for the growth of bacteria. Before starting the generator, the beech shavings should be moistened, and seeded with non-pasteurized vinegar containing acetic acid bacteria. Under these conditions, the filtered fermented juice is run in and allowed to trickle slowly over the shavings. Air enters through the holes in the vat, and passes in the opposite direction to the flow of the liquid. Oxidation or acetification can be com-

pleted by recirculating the liquid through the generator several times. The temperature should be maintained between 80 and 85° F. (27 and 29° C.). Citrus vinegar prepared by the rapid process should be stored, so that esterification and other flavor changes can take place during the aging period. During the slow or Orleans process, esterification usually occurs during the lengthy period of vinegar formation.

From their experimental data, McNary and Dougherty (1960) reported that vinegars of excellent quality and flavor could be prepared from the juices of grapefruit, orange, and tangerine. Their data indicated that the yield of vinegar was formed at a satisfactory rate, which meant that the conversion of alcohol to acid also occurred at a satisfactory rate. Table 182 shows the comparative free-acid contents of various citrus vinegars, prepared from the juices of different species. Grapefruit-juice vinegar was distinguished not only for its high percentage of total acids, but for its exceptionally high citric acid content, which indicated that the acetic acid content had a tendency to be relatively low. In this particular experiment, the final product had 4.08 per cent acetic acid with 1.52 per cent citric acid. These figures indicated that the total acidity of this vinegar could not be reduced below 56 grains without a corresponding reduction in acetic acid below 4 per cent (the minimum legal requirement).

Since sourness is the chief physiological characteristic of vinegar, the citric acid in citrus fruit may not only increase the total acid content of the vinegar made from it, but may act as a valuable dietary constituent. This may indicate certain advantages to be gained by the use of high-acid grapefruit for the production of vinegar.

Orange-peel vinegar which was produced from the press liquor was different from the other types of vinegar in that citric acid was absent from the final product (table 182). The natural fruit acids in the press liquor had been neutralized as a result of the liming process used in producing it from ground peel. In orange-peel vinegar, the sour principle or total acidity of citrus-peel vinegar was attributed almost entirely to the acetic acid present.

TABLE 182

TYPICAL FREE-ACID CONTENTS OF SEVERAL CITRUS VINEGARS.*
(Expressed as percentages.)

Fruit sources	Citric acid	Acetic acid	Total acids
Orange vinegar.....	0.87	4.20	5.07
Grapefruit vinegar....	1.52	4.08	5.60
Tangerine vinegar.....	0.62	4.38	5.00
Orange-peel vinegar...	0.00	5.00	5.00

*From McNary and Dougherty (1960).

Alcohol. Nolte *et al.* (1942*a*) demonstrated that citrus molasses could be used as an economical source for the manufacture of ethyl alcohol. Work in this field began during World War II, when the supply of molasses of other kinds (cane and beet) was not sufficient to supply the demand.

Hendrickson and Kesterson (1951) gave the steps in the manufacture of alcohol from citrus molasses as follows: (1) weighing and mixing, (2) preparation of the pure yeast culture, (3) addition of pure yeast culture, (4) fermentation, and (5) distillation.

Citrus molasses contains carbohydrates in a form which is directly fermented by yeast to ethyl alcohol (C_2H_5OH). Citrus press liquor is too dilute in fermentable sugars to provide a reasonable concentration of alcohol for economical recovery. Consequently, citrus molasses may be added to the press liquor until the sugar content is 10 to 12 per cent. Likewise, citrus molasses may be diluted with water to the desirable concentration. About 25 gallons of press juice are required to make 1 gallon of 190-proof alcohol. Whether alcohol made from citrus can compete with that made from other sources depends on various economic factors. (One gallon equals 3.79 liters.)

Raw materials available for the production of alcohol or alcoholic beverages include (1) starchy substances, such as potatoes, corn, barley, and rice; (2) fruits and fruit juices that contain sufficient concentrations of sugars for fermentation; (3) cane and beet sugars; and finally (4) the molasses derived from processing any of them. It can be observed that some of these materials have high concentrations of fermentable carbohydrates that are capable of being converted to alcohol. Consequently, the production of alcohol from various citrus fractions and components at a reasonable profit depends upon the available quantity of these high-starch and sugar-containing materials at competitive prices.

Yeast. Citrus press liquor is normally used for making molasses for stock feed, but it may also be used for the manufacture of yeast, alcohol, and lactic or butyric acids. Nolte *et al.* (1942*a*) were probably the first to attempt the manufacture of yeasts from citrus press liquor. They used *Torula utilis,* a wild, fast-growing yeast, because it propagated rapidly, produced a relatively small amount of alcohol, and gave a better yield of yeast than any of the other 4 kinds tested. From a liquor containing 1 per cent sugar, they obtained yields ranging from 4.4 to 4.8 per cent dry yeast. Higher yields and faster propagation were obtained when the liquor contained not more than 1 per cent total sugars.

Later, Veldhuis and Gordon (1947) devised a continuous-flow method by means of which they were able to obtain 60 per cent yields when the press liquor was distilled with 2 volumes of water, and 33 per cent yields with full-strength press liquor. This was the equivalent of 20 to 36 pounds of feed yeast per 1,000 pounds of 10° Brix press liquor processed (about 9 to 16 kilo-

grams per 454 kilograms). (See Hendrickson and Kesterson, 1951, for a detailed description of production methods.)

Table 183 was compiled from a paper by Nolte *et al.* (1942*a*), who analyzed a composite of 10 samples. It is generally known that, in addition to the components listed in the table, yeasts contain significant quantities of thiamin, riboflavin, ergosterol, niacin, and pantothenic acid. Table 183 shows that over 50 per cent of the yeast was high-quality protein.

Veldhuis and Gordon (1947) also pointed out that press juice had the advantage of having an abundant supply of carbohydrates which were readily available for the yeast. During passage through the propagators, the yeast utilized about 95 per cent of the sugars in the juice and about 65 per cent of the total organic matter. A 90-pound box of grapefruit yielded about 3 gallons (11.37 liters) of press juice with about 10 per cent soluble solids. Each 3 gallons of press juice produced about 1 pound (0.454 kilogram) of dry yeast.

Hendrickson and Kesterson (1965) noted that yeast had been regarded as a stock-feed supplement because it was rich in B-vitamins and crude protein (about 50 per cent), comparing well with other proteins in digestibility.

Although citrus-feed yeast has not achieved importance as a high-protein feed for animals, it can be refined and made more palatable for human consumption. From a nutritional viewpoint, this rich natural source of biologically complete proteins has many beneficial uses.

Production of Citric and Lactic Acids. Citrus juices contain varying quantities of citric acid, which is one of the most widely distributed organic acids in plant cells. Those fruits that contain higher concentrations of acid, such as lemons, limes, and bergamots (*Citrus bergamia*), are the kinds commonly used in the production of citric acid. At one time in Italy and the United States, lemons were the principal source materials for citric acid. In the West Indies, limes, which usually have a higher acid content than lemons, are used in the manufacture of citric acid.

TABLE 183

COMPOSITION OF DRY YEAST AND ANALYSIS OF ASH.*

Dry yeast	Per cent	Ash	Per cent
Moisture	1.29	Phosphorus pentoxide	45.75
Protein (N × 6.25)	55.28	Magnesium oxide	4.95
Crude fat	4.51	Calcium oxide	1.26
Glycogen	12.78	Silicon dioxide	2.58
Cellulose, gum, etc.	18.06	Sulfur trioxide	7.79
Ash	8.08	Chlorine	0.22
		Iron oxide	0.40
		Sodium and potassium oxides	37.05

*From Nolte *et al.* (1942*a*).

TABLE 184

FERMENTATION OF GRAPEFRUIT JUICE FOR THE PRODUCTION OF LACTIC ACID.*

Experiment numbers	Quantity of juice taken (cc)	Total sugars as invert before fermentation (per cent)	Total sugars as invert after fermentation (per cent)	Sugar consumption (per cent)	Yield of lactic acid (100%) as determined by analysis (per cent)	Fermentation yield of lactic acid (per cent of sugar converted)	USP calcium lactate recovered† (per cent)	Per cent 50% lactic acid recovered†
1.....	200	7.19	1.38	80.8	5.35	74.4	6.0	6.5
2.....	200	7.19	0.40	94.4	6.05	84.1	6.1	6.7
3.....	7,570	7.50	1.79	76.1	5.32	70.9	5.1	...
4.....	15,140	7.48	.93	87.6	5.31	71.0	6.1	...
5.....	75,700	7.28	.60	91.7	5.60	76.9	5.6	8.1

*From Nolte and von Loesecke (1940*d*).
†Based on weight of juice.

Citric acid may be manufactured chemically from lemon juice not used for juice products. The acid in this juice is precipitated as calcium citrate by adding ordinary lime. This citrate, collected in filter presses, may be dried and stored for later conversion into acid, or may be converted directly. Calcium citrate is carefully treated with sulfuric acid to yield a precipitate of gypsum and a weak solution of citric acid. This gypsum is collected in filter presses and discarded. Crystals of citric acid are recovered from the chemically purified solution by multiple-vacuum concentration and crystallization, then are washed, dried, and packed. In the United States, the production of citric acid from the juice of lemons and other citrus fruits has been replaced by the submerged-culture method of Karow and Waksman (1947).

Nolte and von Loesecke (1940*d*) described a method for preparing calcium lactate and lactic acid from grapefruit by fermenting the screened juice with a lactic acid bacterium of the *Lactobacillus delbruckii* type, normally present on the fruit. Grapefruit juice was fermented by the addition of a starter culture of lactic acid bacilli, with the temperature maintained at 45 to 55° C. (113 to 131° F.). By keeping the temperature within these limits, the danger of alcoholic or butyric acid fermentation was avoided. Because the lactic acid bacillus was very sensitive toward free acidity (over 1.5 per cent concentration), the fermentation tended to cease after a short time. To prevent this, the lactic acid was neutralized by adding calcium carbonate at the beginning of the operation, thus obtaining lactic acid in the form of its sparingly soluble calcium salt. This salt was then treated with dilute sulfuric acid, and free lactic acid was obtained from the filtrate by distillation *in vacuo*. Lactic acid could be recovered either as calcium lactate or as actual lactic acid. In these experiments, the yields of lactic acid using grapefruit juice as the substrate varied from 71 to 84.1 per cent of the soluble sugars converted during fermentation. The amounts of sugar consumed in the process ranged from 76.1 to 94.4 per cent of the total soluble carbohydrates (table 184).

Naringin (Flavonoid)

CITRUS FRUITS are comparatively rich in flavonoid constituents; the two that have the most economic potential are hesperidin, which occurs in sweet, sour, and mandarin oranges, and naringin, which is the predominant flavonoid in grapefruit and pummelo (Horowitz, 1961). Since naringin is the chief bitter principle of grapefruit juice, its physical and chemical properties have been studied in considerable detail, and the results have been discussed in chapters 2, 3, and 4.

Naringin is a flavanone glycoside extracted from grapefruit peel, and is used commercially to impart a desirable bitterness to beverages, confections, and marmalades made from sweet oranges. Pulley and von Loesecke (1939) suggested that naringin be used as a raw material for the production of

rhamnose. Hendrickson and Kesterson (1956) obtained a patent for the production of azo dyes from naringin. For many years, naringin was produced in the United States in limited amounts, and was sold under various trade names. It has no known therapeutic properties.

Grapefruit Seed By-Products

DUNCAN GRAPEFRUIT is a seedy variety of citrus fruit that contains approximately 4.4 per cent seeds, based on the weight of the whole fruit. In those areas where large volumes of this seedy grapefruit are grown and processed, a plentiful supply of seeds is available for oil extraction. It so happens that Florida is not only the largest grapefruit-producing area in the United States, it is also the largest grapefruit-processing area. Consequently, research and development projects on the conversion of citrus seeds to by-products have received considerable attention by scientists in both the United States Department of Agriculture and the Florida Agricultural Experiment Station (USDA Handbook 98, 1956; Hendrickson and Kesterson, 1965).

Physical and Chemical Properties of Seeds in Relation to Maturity. According to the data of Hendrickson and Kesterson (1965), the most abundant sources of seeds are first, Duncan grapefruit, and secondly, the Pineapple and Parson Brown orange varieties (table 185). In Florida, these 3 varieties are the commercial sources of raw seed for citrus-seed oils. Since fruit weight increases during the season, the percentage yield of seed calculated on the fresh-weight basis would be significantly affected by the rate of change in fruit maturity. The rate of change in fruit size is highly correlated with the rate of fruit growth, and consequently with the rate of change in fruit weight. Citrus seeds of all varieties may not only decrease in moisture with fruit maturity, but may become green in color in overripe fruit, and sprout while they are still enclosed in the fruit. Data are also reported on the percentages of seeds and seed oil potentially available in dried citrus pulp obtained from residues of different types of citrus fruits. It follows that seasonal changes affecting growth and maturity of the fruit will affect the seed maturity, which in turn will affect the percentage of oil taken from dried citrus seeds.

The physical and chemical properties of citrus-seed oils have been reported in chapter 3, but the important subject of utilization of grapefruit seeds for the production of oil and other by-products remains to be discussed.

Grapefruit seeds are ejected with the pulp during the processes of canning the juice and segments, and the fresh seeds are separated from the adhering pulp and rag by passing the ejected residue through a rotating reel with a coarse screen. The seeds are coated with a highly polymerized and methoxylated galacturonide that chemically resembles pectin, but which, unlike pectin, fails to produce characteristic sugar jellies. The seeds may be separated from the pulp and rag with a paddle-type finisher. Another method is to

treat the mixture of seeds and wet pulp with lime slurry, and afterward dry the treated material in rotary driers. The seeds can easily be separated from the dried pulp by screening, and the pulp mixed with the dried citrus feed.

Proximate Analyses of Seeds. The analyses of whole, air-dried grapefruit seeds and air-dried hulls, which are the outer seed coats, are compared in table 186. The crude fat or the ether-soluble extract of the whole seeds amounted to 30.30 per cent, while in the hulls, the ether extract amounted to only 1.17 per cent. This relationship would be expected to exist, because the oil occurred chiefly in the seed embryos. The protein fraction amounted

TABLE 185

SEED AND OIL DATA OF SEVERAL VARIETIES OF ORANGES AND GRAPEFRUIT BEFORE AND AFTER PROCESSING TO DRIED PULP.*

(Data are stated as percentages.)

Fruit types	Fresh fruit		Dried citrus pulp (8 per cent water)	
	Seeds	Moisture in seeds	Seeds	Seed oil
Hamlin orange	0.45	56	2.4	0.9
Parson Brown orange	2.3	54	12.2	4.5
Pineapple orange	2.7	50	14.3	5.3
Valencia orange	0.8	48	4.3	1.6
Duncan grapefruit	4.4	58	17.7	5.4
Marsh grapefruit	0.25	56	1.0	0.3

*From Hendrickson and Kesterson (1965).

TABLE 186

ANALYSIS OF WHOLE, AIR-DRIED GRAPEFRUIT SEEDS AND AIR-DRIED HULLS.*

Seeds (per cent)		Hulls (per cent)†	
Moisture	11.86	Moisture	10.19
Crude fat (ether extract)	30.30	Ash	2.97
Protein (N × 6.25)	15.94	Protein (N × 6.25)	4.00
Crude fiber	9.14	Crude fat (ether extract)	1.17
Ash	2.48	Crude fiber	39.75
Nitrogen-free extract (by difference)	30.28	Cellulose	32.50
SiO_2	0.28	Pentosans	16.34
$Fe + Al(Fe_2O_3 + Al_2O_3)$	.50		
Ca	.36		
Phosphates (P_2O_5)	.56		
Na	.052		
K	.54		

*From Nolte and von Loesecke (1940*b*).
†Hulls constitute approximately 23 per cent of the air-dried seeds.

to 15.94 per cent of the weight of the air-dried seeds, and to 4 per cent of the air-dried hulls. The nitrogen-free extract in the whole seeds amounted to 30.28 per cent on the air-dried basis, and this value was the percentage of residue left after subtracting the sum of the percentages of protein, ether extract, ash, crude fiber, and moisture from 100 per cent. Since this fraction was determined by difference, it represented roughly the total carbohydrate present. The hulls were noted for a large amount of crude fiber (39.75 per cent), which was an empirical value. It is not in any sense a chemical compound, but is a mixture of substances which make up the structural polysaccharides of the tissues, such as cellulose, lignin, and some hemicellulosic materials (pentosans). The relatively low concentration of crude fiber that existed in the seeds (9.14 per cent) was contrasted with the high quantities of fats, carbohydrates, and proteins present in the endosperm and embryo.

Ammerman *et al.* (1963) reported additional data on the composition of citrus seeds, determined on samples composed of mixtures of both grapefruit and orange seeds (table 187). These seeds yielded, on the average, 26.5 per cent hulls and 73.5 per cent kernels, calculated on the dry-weight basis of the whole seeds. The proximate compositions of the whole seeds, hulls, and kernels (seeds minus hulls) are reported on the dry-weight bases of the respective samples in columns 2, 3, and 4 of the table. Similar to the values reported on grapefruit seeds (table 186), the amounts of crude fat (ether-soluble extract) were exceptionally high in whole seeds and kernels, and were exceptionally low in the hull fraction. The same relationship held with respect to the protein contents of the various components. Of the 3 components, the hulls contained the highest quantities of crude fiber (48.1 per cent) and nitrogen-free extract (39.1 per cent), and the whole seeds contained greater amounts of these constituents than the kernels. When calculations were reported on a hull-free basis, the protein content of the whole seed

TABLE 187

AVERAGE COMPOSITION OF CITRUS SEEDS, SEED HULLS, AND KERNELS.*

Factors	Seed components (per cent)				
	Whole	Hull	Kernel	Extracted whole†	Extracted kernel†
Moisture	0.0	0.0	0.0	10.0	10.0
Crude fat (ether extract)	45.1	2.3	59.2	0.0	0.0
Protein (N × 6.25)	16.2	6.1	19.5	26.5	43.0
Crude fiber	13.2	48.1	3.1	22.1	6.8
Ash	3.4	4.4	2.8	5.6	6.2
Nitrogen-free extract	22.1	39.1	15.4	35.8	34.0

*Data of Ammerman *et al.* (1963), tabulated by Hendrickson and Kesterson (1965).
†Calculated.

was increased from 16.2 to 19.5 per cent in the kernel, and the crude fiber was reduced from 13.2 to 3.1 per cent. Other calculations made on a fat-free basis increased the protein from 16.2 per cent in the whole seeds to 26.5 per cent in the extracted whole seeds, and the crude fiber increased from 13.2 to 22.1 per cent. By calculating the various components on a hull and fat-free basis, the kernels contained 43 per cent protein, 6.8 per cent crude fiber, and 34 per cent nitrogen-free extract (column 6, table 187).

Seed Oil. Dried seeds are the raw materials from which the oil is expressed. Seeds, with or without hulls, may be passed through a roller-milling process which mechanically cracks and breaks them so the hulls (if present) can be removed by screening, and the oil can subsequently be extracted from the kernels or whole seeds at high pressure in an oil expeller at elevated temperatures. The expressed oil is forced through a plate-and-frame press for clarification.

The crude product that is commercially produced from grapefruit seeds has a bitter flavor, and the source of this bitterness has been attributed to limonin ($C_{26}H_{30}O_8$) isolated from the soap stock obtained in refining the crude oil (Nolte and von Loesecke, 1940*b*). This intensely bitter compound is a dilactone, which is easily converted by alkali to a salt of limonic acid which is not bitter, but acidification causes cyclization, regenerating limonin with its original bitterness. Although the chemical and physical properties of the two oils are somewhat different, no attempt is made in Florida to separate the predominant amount of grapefruit seeds from the smaller amounts of orange seeds prior to extraction (Van Atta and Dietrich, 1944; Nolte and von Loesecke, 1940*b*).

The 3 products of citrus seeds that have commercial importance are the oil, meal, and hulls. The oil can be expressed or extracted by solvents from dried citrus seeds. The seed oil of commerce is usually produced by pressure methods. After the freshly expressed oil has been transported to settling tanks for a brief period, it is pumped through filters into storage tanks, where final settling occurs. If the oil is to be used for edible purposes, it is refined by treating with alkali, which removes the limonin and free fatty acids. The neutral oil resulting from this refinement may be hydrogenated for solidification and thus used for spreads or for shortening, or it may be winterized by chilling to crystallize the higher-melting glycerides, which can be removed by filtration. The object of winterization is to produce oil which will not solidify when stored at refrigeration temperatures between 40 and 45° F. (4.4 and 7.2° C.). Such oil is suitable for use in the production of salads and mayonnaise. According to Nolte and von Loesecke (1940*b*), unrefined grapefruit-seed oil will solidify between 10 and 14° F. (–12 and –10° C.). For nonedible purposes, the unrefined citrus-seed oils can be used in those situations where semidrying oils are required.

Hendrickson and Kesterson (1965) reported that brominated citrus-seed oils with relatively high specific gravity were being used to adjust the density of flavoring oils used in beverages. Brominated citrus-seed oil was found to be equal in quality to other brominated products, such as apricot-kernel oil and sesame oil.

Seed Meal and Hulls. Since citrus seeds are processed chiefly for oil, seed meal can be made from the residue left after pressure extraction (also known as press cake), or it can be the residue after the removal of both hulls and oil from the seeds. Both types of citrus-seed meal have been used in animal nutrition experiments.

Glasscock *et al.* (1950) reported that citrus-seed meal (press cake) was just as valuable as cottonseed meal in meeting the protein requirements of growing and fattening steers, and that it was readily consumed by the steers with no indications of toxicity. Similar experiments with pigs showed that citrus-seed meal (expeller-processed) was unpalatable and harmful when fed at levels as low as 10 per cent of the total ration, and consequently should not be used as a dietary supplement for them.

Driggers *et al.* (1951) reported that dried citrus-seed meal was toxic to chicks, and that the toxic fraction (limonin, the bitter principle of citrus seeds) could be removed by extracting the meal with diethyl ether, acetone, and 95 per cent ethyl alcohol. The detoxified citrus-seed meal could be used in the chicks' diet up to 20 per cent of the total ration and, at this percentage level, it was equivalent to soybean meal as a source of vegetable protein.

In Florida, citrus-seed meal is mixed with dried citrus pulp, which is used chiefly as a cattle feed. When citrus seeds are processed for commercial oil, there remains a residue which can either be returned to the dried pulp or separated into hulls and citrus-seed meal. Since citrus-seed oil has a higher feeding value than the carbohydrates of the dried pulp, those lots of dried pulp which contain seeds have higher nutritive value than similar products containing no seeds.

Grapefruit-seed hulls have a relatively low feed value in that they contain small quantities of fat and digestible protein. The hulls have been used for soil conditioning in the same manner as those of cotton seeds, tung, rice, peanuts, etc. According to Ammerman *et al.* (1963), citrus-seed hulls are equal in feeding value to cottonseed hulls as determined by livestock-feeding trials.

Citrus Waste Disposal

WASTE DISPOSAL and by-product utilization have continued to grow in importance as a result of the continued increase in the amount of citrus fruit processed. Solid wastes from citrus-juice processing plants amount to millions of tons annually. Consequently, tremendous quantities of liquid effluents from processing plants must also be disposed of without creating health haz-

ards. The object of purification of these waste liquors is primarily to obtain an effluent which will not pollute the watercourse into which it is finally discharged. An effluent may be said to be entirely satisfactory if it avoids secondary decomposition which may give rise to aerial pollution, and will not cause de-aeration of the body of water into which it is discharged. These deductions are based on the fundamental generalization that biochemical reactions in natural waters are highly correlated with changes in the amount of dissolved oxygen. Oxygen therefore plays a most important role in the decomposition process of organic materials, and the amount of oxygen dissolved in the water is highly important in maintaining a standard of reference for the quantity of pollutants or oxygen-demanding constituents that may be discharged into a watercourse. It thus follows that when oxygen in the water is consumed by decomposing polluting materials, fish and plant life die, resulting in anaerobic or putrefactive reactions which have the potential of creating a major nuisance. Consequently, the allowable loading is estimated from the quantity and oxygen demand of the citrus waste water. By the application of these principles, the necessary treatment can be given to the waste liquid to reduce the load of pollutants that enters the receiving stream to a proper level.

From the data reported in this chapter, it can be observed that considerable progress has been made in the disposition of both solid and liquid wastes that have resulted from the extraction of juice from citrus fruits. The solid waste is composed chiefly of peels, cores, rag, and seeds. It is this fraction from which most of the by-products are produced. The liquid or soluble wastes consist chiefly of cooling waters, floor washings, waste waters from peeling and sectioning tables, fruit washers, spent lye, and other waste materials. Depending, of course, on the concentration of soluble organic constituents, liquid waste requires a high biochemical oxygen demand (BOD), and it is of considerable importance in environmental pollution to dispose of this material without creation of a public health hazard. Differences in the oxidizing rates of various waste materials under a given set of conditions, using the same seed material, can chiefly be attributed to differences in chemical composition as related to metabolic activities of the microorganisms.

Biological Oxidation of Citrus Waste. Biological treatments that have been used to dispose of citrus waste waters are yeast fermentation, methane production, trickling filters, and an activated sludge process.

The fermentation of citrus waste liquors by yeast is an old and established method for removing soluble carbohydrates from effluents which usually constitute approximately two-thirds of the total organic matter. After fermentation, the organic matter that remains includes alcohol, esters, pectic substances of various molecular sizes, glycosides, and salts of citric acid (Veldhuis and Gordon, 1947).

Von Loesecke *et al.* (1941) were among the earlier workers who reported on the purification of cannery waste by various methods, such as chemical precipitation, activated sludge, contact filters, and sand filters, and they concluded that trickling filtration was the most promising procedure for the disposition of cannery effluents. They suggested a formula for deriving the size of the filter, which included such variables as the quantity of effluent to be treated and its BOD. Although these factors varied with nearly every cannery, the average pounds of BOD removed per cubic foot[4] of filter proved to be 0.041. The efficiency of the filter seemed to increase with an increase in temperature.

McNary *et al.* (1951) produced methane by anaerobic fermentation of citrus waste water. The fermentation reaction proceeded satisfactorily except in the presence of citrus-peel oils, which proved to be toxic to the anaerobic bacteria. Complete removal of the essential oils from citrus liquors was obtained by permitting fermentation to take place with naturally occurring yeast during the time the liquor was being vigorously aerated. The volume and concentration of these wastes were such that lime had to be added to maintain a pH of about 7.0 if the solids were below 1 per cent.

In studying the disposal of citrus-processing wastes, Wakefield (1953) applied 4 methods that were in use in treating sewage to the treatment of citrus waste liquids. High-rate trickling filters yielded satisfactory results which could be predicted with reasonable organic loading of the filter surface. These filters removed, on the average, 0.182 pound of BOD per cubic foot of filter volume. (See the footnote below.) This amount was the equivalent of 76 per cent of the applied BOD, which totaled 0.239 pound per cubic foot of filter volume. The most efficient performance occurred in those filters where 50 ppm of nitrogen and 20 ppm of phosphorus were added to the raw waste. The trickling of the waste liquor over the limestone and slag filters brought about a close contact among the organisms, oxygen, and organic constituents dissolved or finely suspended in the applied waste water. The biological oxidation of this waste converted much of the organic matter to forms that could settle and be removed. The recirculation of the effluent through the unit to increase the supply of organisms and oxygen resulted in a higher efficiency per unit of filter surface.

Oxidation of waste liquor solids by activated sludge can be performed in aeration tanks in which compressed air is released at the bottom of the tanks in order to supply an ample amount of oxygen and suitable agitation of the contents. The sludge that is produced can be removed to a settling tank, and part of it can be recycled through the system to renew the supply of organisms and to create a large surface for the absorption of incoming solids.

[4] One pound equals 0.454 kilogram; hence, 0.041 pound equals about 0.019 kilogram. One cubic foot equals 28.3 cubic decimeters.

According to Wakefield (1953), the activated sludge process held considerable promise for the oxidation of solids in citrus waste waters. His data indicated that 7 out of 10 operating conditions resulted in 80 per cent reduction in BOD. During this experiment, 22 cubic feet of air per gallon (3.79 liters) of waste were required to maintain 2 to 3 ppm of dissolved oxygen (DO) in the effluent.

Dougherty *et al.* (1955) experimented with the activated sludge process, using a laboratory model for the treatment of citrus waste liquors. Orange juice containing varying amounts of pulp (chiefly juice sacs) was the test material. Since the juice vesicles consisted chiefly of cellulose, the latter was a stable carbohydrate that was not greatly changed during the short detention time in the aeration chambers. They observed that the BOD of the waste liquors was highly related to the oxidized dissolved and colloidal materials which were correlated with the substances most easily changed during this type of treatment. As a result of these experiments, a high-quality effluent was produced that was clear and odorless, and resulted in the removal of more than 90 per cent of the BOD of the raw waste. The dried sludge produced from citrus waste liquors was dried and used as a source of B-vitamins in animal-feeding trials.

In Florida, Wakefield (1953) reported that the use of spray irrigation to dispose of citrus waste on pasture and wooded land had proved destructive to vegetation. Soils that had a high initial capacity to absorb citrus waste liquors showed a significant rate of decrease as the soil became saturated. Soil covered with vegetation or heavy mulch showed greater capacity to absorb the waste than did barren land. Since spray irrigation was considered to be a waste-disposal method, it was most easily adapted to plants located in areas of high, dry land which were inexpensive to buy or rent.

Wakefield also noted that chemical flocculation, followed by aeration to promote agitation and floating of the particles, did not prove to be exceptionally efficient. The flocculation of citrus waste liquor with lime removed, on the average, 61 per cent of the total suspended solids during an 8-day testing period. A settling experiment on the composited raw samples resulted in removing 64 per cent of the suspended solids. The flocculation treatment of citrus waste did not significantly reduce the oxidation demand of the waste, because the most highly oxidizable substances were found to remain in soluble forms in the waste liquor.

Chapter 7

Grapefruit Processing: General Methods

MUCH OF THE GRAPEFRUIT produced in this and other countries is processed as juice or segments. Juice may be canned as single-strength, frozen concentrate, or nonfrozen concentrate. Segments are usually canned, but some were once marketed frozen in paper packages. Most citrus waste from processing—peel, rag, and seeds—is used to make such by-products as stock feed, molasses, pectin, peel oil, seed oil, marmalade, and alcohol. These products have been discussed in chapter 6.

The development of some of the special methods necessary for the processing of grapefruit is reviewed in this chapter, but the steps used in processing are described only briefly and generally. It would take us far afield to discuss in detail all the commercial procedures that have to do with mechanical handling of the fruit, transfer of fluids and other fruit components, as well as preparation and separation of the fruit and its constituents, including filtration, evaporation, sterilization, and drying of various citrus fractions. For detailed descriptions of such commercial methods for the various products, see Mottern and von Loesecke (1933), Shearon and Burdick (1948), Braverman (1949), Veldhuis (1951), Hendrickson and Kesterson (1951), Tressler and Joslyn (1954), Joslyn and Heid (1963), Heid and Joslyn (1963), and others who may be cited in this chapter.

Blended Citrus Juices

BLENDED JUICES composed of equal parts of orange and grapefruit juice were first processed and packed in Florida in 1935–36. In present-day practice, the ratio of orange to grapefruit juice used in producing this blend is highly associated with the color desired, the acid concentration, and the amounts of soluble solids in both juices. Most orange–grapefruit blends contain 50 per cent orange juice, but when it is light in color, the concentration may go as high as 75 per cent orange juice. According to federal standards, the grade A blend must contain not more than 12 per cent free and suspended pulp and not more than 0.030 per cent by volume of recoverable oil. It must be free of seeds and seed fragments as well as other substances that would affect the appearance of the product.

Grapefruit Segments

THE FIRST CANNING of grapefruit segments in the United States on a commercial basis occurred in Florida about 1919 or 1920, according to Stevenson (1934). In fact, canned grapefruit segments were among the first successful citrus products, and before 1936 the volume of segments processed exceeded that of any other canned citrus product. Stevenson noted that canned segments had a good storage life, and were popular ingredients of fruit cocktails, salads, and desserts. At one time, frozen segments were packed in paper containers instead of in cans, but were not so popular because they had to be thawed before being used. Veldhuis (1951) described the methods for processing both canned and frozen segments.

Duncan grapefruit, a seedy variety, is by far the most prominent citrus fruit used in Florida for the canning of segments, and the Marsh variety, a seedless type, is the one that is similarly used in Israel. At the present time, segments account for approximately 20 per cent of all the grapefruit processed in Florida. According to the observations of Singleton (1956), Marsh Seedless grapefruit was formerly preferred for sectioning when peeling was done solely by hand, but with the introduction of alkali in the peeling process, the seeded fruit became preferable to Marsh Seedless because of its solid core. Lye penetrated the hollow core of the Marsh variety and destroyed the membranes which bound the carpels together, thus yielding fragile segments which fell apart when the fruit was handled.

Sectionizing Procedure. Grapefruit segments are prepared from the endocarp of the fruit by separating the unbroken juice vesicles from the segments by removing the carpellary membrane. The procedure for processing and canning grapefruit segments begins with fresh fruit of good internal quality. When grapefruit is delivered to the processing plant, it is graded and sized. All fruits too large or too small to be used for sectionizing go to the juice plants. Uniformity of segments in the canned product is controlled by using fruit of uniform size. The fruit is washed, and then passed on conveyors through a steam chamber, or placed in water at approximately 196° F. (91° C.) for about 5 minutes, which loosens the peel from the edible pulp. The fruit is next cooled by sprays of cold water as it emerges from the scalding tanks; the peel is then removed by hand. The peeled fruits are passed through a 2 per cent lye solution for approximately 30 seconds to remove the remaining albedo and segment membranes. After this treatment, the lye is removed from the fruit surfaces by again spraying with water. The sections can be removed by hand with a triangular spatula, or by using an automatic sectionizer. The segments are packed in syrup in cans which are heated to remove air, sealed, pasteurized in hot water, and cooled. After removal of the carpellary membranes, the individual segments are fragile, and care has to be taken

to prevent the whole segment from separating into smaller aggregations of juice vesicles.

From the foregoing description, it can be seen that in the past, the processing of grapefruit into canned segments was accomplished chiefly by manual labor. This is one area of citrus processing that has needed a research program for its development; specifically, processing machinery that would peel, sectionize, and pack fresh grapefruit segments. The structural components of grapefruit, such as peel, segments, segment membranes, and juice vesicles, are separated within the fruit into compartments to such an extent that automatic machinery should be highly adapted for the separation of the segments from the other tissues. The manual methods of peeling and sectionizing grapefruit are slow and expensive; consequently, a continuous process of mechanization would not only result in increased saving in labor cost, but would raise the level of productivity enormously. To make possible the economical production of more and better segments, automated equipment has been developed that can peel, sectionize, and pack grapefruit segments at relatively high speed.[1]

The citrus peeler (FMC unit) removes the peel from grapefruit or oranges at the rate of approximately 40 fruits per minute. This automated machine is constructed with 8 fruit-holder cups mounted on a revolving table. Each fruit completes a full revolution of the table, passing through 8 different operational "stations" before being ejected, completely peeled. The fruit is fed to the machine from a conveyor through an automatic feeder which deposits 1 fruit in each cup as it passes the feeder station. The sequence of operations follows, including the initial, or "feeder," one.

1. The rotation of the turntable is synchronized with a feed mechanism which regulates the deposit of 1 fruit in each cup as it passes the feeder station.
2. The operator centers the stem end of each fruit under a fine beam of light.
3. A hole is cut through the stem end of the peel by an auger that rotates inside a cylindrical tube.
4. Eight simultaneous cuts, equally spaced, are made through the peel, starting at the hole just drilled in the stem end and extending down the side past the equator of the fruit.
5. At this opener station, 8 fingers enter the hole in the peel and move rapidly outward and upward, lifting the tip of each peel segment.
6. At this stripper station, 8 strippers clamp the segment tips and, by a downward-rolling action over the surface of the peel, pull it outward and downward.
7. Eight fingers slide the peel down to the cup, where it is clamped by a ring on which the fingers are mounted. When the peel is clamped down, a wiper removes the peeled fruit and ejects it onto a take-away conveyor.
8. At this final station, a rotating nylon-tufted brush sweeps the peel from the cup. The empty cup then proceeds to the feeder station for a repetition of the cycle.

[1] The Food Machinery Corporation (Florida Division) has supplied the author with generous information on their automatic *Citrus Peeler* and *Citrus Sectionizer* through the good offices of W. C. Belk, Chief Engineer.

The citrus sectionizer (FMC unit) now takes over where the peeler unit has finished its cycle. The peeled fruits are placed manually at about 30 per minute into a cup on the feeder table. The operator places the fruit blossom end up, centering the end under a small beam of light. The blossom end is kept on top because the partitions between sections are more mature and are stronger than on the stem end. Precision devices now complete the accurate centering of the fruit, which proceeds through the 12 operational stations described here, supported by an inflated latex cushion.

1. A transfer mechanism moves the fruit to a spindle at this feeder station, with the center of its core precisely aligned with the center of the spindle.

2, 3. Right-hand and left-hand pathmaker mechanisms here provide paths in the fruit in which probes will be able to seek partitions between the sections.

4 through 9. The fruit now moves through these 6 blade-head stations. The first 3 seek the partitions on the right-hand sides of the sections, and the other 3 seek those on the left-hand sides. Each of these blade-head stations is provided with 5 blades. These blade-head assemblies also have probes which guide the blades to the partitions. A highly complex mechanical system within the assembly permits a probe for each individual blade to seek a partition independently. When the probes are aligned with the partitions, they lock in place and move down through the fruit, separating the sections from the partitions.

10. At this seed-shaker station, probes are inserted into the seed area, where they vigorously shake the sections back and forth to loosen the seeds and separate the sections from the core.

11. A spinner device that rotates about one and one-half revolution positively separates any remaining sections from the partitions or core, and wraps the partitions tightly around the spindle. The sections drop onto a belt and are conveyed to the can or jar fillers.

12. This core-removal station separates the tightly wrapped core from the spindle; the useless core drops into a chute and is carried away.

Chemical and Physical Factors That Affect the Quality of Canned Grapefruit Sections. Since 50 per cent or more of Florida grapefruit is converted into processed products, it is important that data should be obtained on the factors that affect the internal quality so essential for the production of processed commodities of high quality. Consumers of fresh grapefruit relate quality largely to its external appearance and to the minimum maturity standards as measured by the Brix:acid ratio. The processor is more concerned with the total soluble constituents of the juice than with the external appearance of the fruit. In canning unsweetened grapefruit juice, the fruit that is used should contain juice with low acid content and high Brix:acid ratio; in processing sweetened juices, fruit with higher acid concentration in the juice may be processed into products which require the addition of calculated amounts of sugar in order to obtain the desired flavor.

In canning grapefruit sections, a high Brix content of the raw grapefruit is directly related to the quality (or grade) of the product by making the sec-

tions firmer, as well as by decreasing the drained weight loss during and after processing (Carter, 1964).

Wenzel and Moore (1956) correlated the quality of processed grapefruit products with the large variations in the internal quality of grapefruit that had been shown to exist throughout the packing season.

Seasonal and growth changes in fruit exist as a result of many factors, such as differences in varieties, maturity, cultural practices, rootstock, soil, weather conditions, and many other influences, all of which have been discussed at some length in other chapters of this book. Wenzel and Moore emphasized the importance of harvesting the fruit at optimum maturity and at the Brix: acid ratio desirable for the specific product contemplated. Methods are available which, when applied, will produce grapefruit with a low acid content and of high Brix:acid ratio, which is desirable for producing processed products of high quality, and especially those that will not need the addition of sugar. Arsenation is the principal treatment that is used to reduce the concentration of free acidity in the juice and thereby increase the Brix:acid ratio.

Huggart *et al.* (1957*a*) attempted to establish a possible relationship between date of packing and quality factors (flavor, firmness, and color) of commercially canned grapefruit sections during storage at 90° F. (32° C.) for 6 months. Based on monthly examinations, their data indicated considerable variability between samples collected in different seasons. In the 1953–54 season, the data showed that the packing date affected the quality after storage of the samples. In the samples collected from 2 other plants during the 1954–55 season, the data revealed no correlation between the processing date and retention of quality in the stored sections.

In studying the effects of storage temperatures on the quality of canned grapefruit sections, Huggart *et al.* (1955) reported that their good quality could be retained for a year at storage temperatures of 70° F. or lower. After 3 months of storage at 90° F., the flavor of the canned sections proved to be significantly different from that of the control samples stored at 32° F.; detectable changes in color and firmness appeared after 3 to 6 months in storage. At 80° F., grapefruit sections maintained their original quality for 3 to 6 months, but showed notable changes in color, firmness, and flavor after 12 months in storage. (21, 32, zero, and 27° C., in that order.)

It was further demonstrated that browning or discoloration of canned grapefruit sections during storage was associated with the acidity in the canned product, which in turn bore a definite relation to the acid content of the fruit used (Huggart *et al.*, 1957*b*).

It is not to be said that a high hydrogen ion concentration is the sole factor that may influence the browning of grapefruit sections, but discoloration has occurred more frequently in sections with a high acid content. Owing to the high degree of variability in the samples, browning of grapefruit sections in

a pack requires a fairly large sample (number of cans) in order to be able to predict discoloration from the titratable acidity.

Since fertilization of citrus trees is known to affect the physical and chemical properties of the fruit, it follows that fertilizer treatments of grapefruit trees should influence similar characteristics in the canned sections. This generalization was borne out by the data of Wenzel *et al.* (1956*a*), who showed that commercially canned grapefruit sections, processed from fruit picked at the same time from plots that were treated with fertilizers containing 0, 3, and 10 per cent potash, decreased in firmness with an increase in the amount of potash used (table 188). In the 1955 experiments, all the sections in the 3 packs were rated poor in quality because of the high percentages of soft ones, attributed to the fact that the fruit was picked late in the season. Canned sections of high quality cannot be processed from grapefruit that has remained on the trees beyond its prime condition. In the 1956 packs, the number of whole sections amounted to 60, 50, and 30 per cent in fruit from the plots that had received 0, 3, and 10 per cent potash, respectively. In the same samples, the number of soft sections increased accordingly to 11, 36, and 51 per cent. These data followed the same trend as those reported for the 1954 packs. The total acids in the drained syrup increased in concentration as the amount of potash in the fertilizer applied was increased. These results agreed with those of Sites (1950*b*), who reported that the titratable acidity of the juice increased, and the Brix:acid ratio decreased, in grapefruit produced by trees supplied with potash ranging up to 10 per cent of the fertilizer mixture.

Wenzel *et al.* (1956*a*), using fruit samples from the same plots, determined the effects of varying the potash in the fertilizer on the pectic substances in canned grapefruit sections. Three pectic fractions—water-soluble, oxalate-soluble, and sodium hydroxide-soluble—were determined on the whole, broken, and soft sections from the 1956 pack. The data in table 189 show that in all 3 types of grapefruit sections, the water-soluble pectic materials were greater in the fruit from plots receiving the 10 per cent potash level than in that from plots where no potash was present in the fertilizer. The oxalate-soluble pectic fraction varied inversely with the potash level. This can be exemplified by the fact that at the 10 per cent potash level, the whole, broken, and soft sections, respectively, contained 34.3, 35.9, and 36.1 per cent less overall total pectic substances than similar samples from plots with no potash. The soft sections had the most water-soluble and total pectic constituents.

Chilled Grapefruit Juice

THE PRODUCTION of chilled citrus juices has continuously increased with the concomitant increase in consumer demand. Chilled grapefruit juice is marketed in considerable quantities, but the amounts popularly consumed are far less than those of chilled orange juice. In order to increase the use of chilled

TABLE 188

EFFECTS OF VARIABLE POTASH FERTILIZATION ON THE FIRMNESS OF CANNED GRAPEFRUIT SECTIONS.*

Variations of potash in fertilizer (percentages used)	Number of 303 cans examined	Number of sections examined	"Thread" present (per cent)	Firmness (per cent)			Final ratings	Total acid, as anhydrous citric, in drained syrup (per cent)
				Whole	Broken on handling†	Soft		
PACKED FEBRUARY 12, 1954								
K_2O—0	83	1,190	79	68	26	6	Good	0.82
K_2O—3	107	1,659	78	48	33	19	Fair	1.07
K_2O—10	95	1,594	64	28	30	42	Poor	1.25
PACKED FEBRUARY 26, 1955								
K_2O—0	64	1,147	86	47	17	36	Poor	1.00
K_2O—3	66	1,227	86	47	21	32	Poor	1.08
K_2O—10	62	1,204	83	39	16	45	Poor	1.22
PACKED JANUARY 14, 1956								
K_2O—0	47	769	95	60	29	11	Good	0.87
K_2O—3	44	734	90	50	14	36	Poor	1.19
K_2O—10	41	729	85	30	19	51	Poor	1.21
COMMERCIAL PACKS (FOR COMPARISON)								
Seasons packed:								
1953–54	36	652	57	51	48	1	Good	0.94
1954–55	48	876	89	42	51	7	Good	1.01
1955–56	16	286	92	75	21	4	Good	1.05

*From Wenzel *et al.* (1956*a*).
†These sections were whole when removed from cans to screens, but they broke into two pieces upon further handling during examination.

TABLE 189

EFFECTS OF VARIABLE POTASH FERTILIZATION ON THE PECTIC SUBSTANCES IN CANNED GRAPEFRUIT SECTIONS.*

Variations of potash in fertilizer (percentages used)	Whole sections		Broken sections		Soft sections		Total pack
	Per cent of 1956 pack	Pectic substances (mg/100 g)	Per cent of 1956 pack	Pectic substances (mg/100 g)	Per cent of 1956 pack	Pectic substances (mg/100 g)	Pectic substances† (mg/100 g)
WATER-SOLUBLE PECTIC SUBSTANCES							
K_2O—0	60	80	29	90	11	109	86
K_2O—3	50	80	14	95	36	104	90
K_2O—10	30	87	19	102	51	129	111
AMMONIUM OXALATE-SOLUBLE PECTIC SUBSTANCES							
K_2O—0	60	257	29	262	11	272	260
K_2O—3	50	141	14	146	36	160	149
K_2O—10	30	131	19	121	51	126	127
SODIUM HYDROXIDE-SOLUBLE PECTIC SUBSTANCES							
K_2O—0	60	158	29	163	11	170	161
K_2O—3	50	97	14	107	36	107	102
K_2O—10	30	107	19	107	51	97	102
TOTAL PECTIC SUBSTANCES							
K_2O—0	60	495	29	515	11	551	507
K_2O—3	50	318	14	348	36	371	341
K_2O—10	30	325	19	330	51	352	340

*From Wenzel *et al.* (1956*a*).
†Calculated values based on the percentage of whole, broken, and soft sections in each pack.

grapefruit juice, it is essential to improve the quality of the product from the standpoints of both flavor and soluble solids.

Wenzel *et al.* (1956*b*) presented a summary of the important factors, such as fruit quality, concentrate quality, processing procedures, plant sanitation, and storage temperatures, that influence the quality of chilled citrus products. These same factors are equally important in the production of frozen citrus concentrates. Chilled citrus juices should have as nearly as possible the same physical and chemical properties as the freshly extracted juices. Without considering the early developments of chilled citrus juices in sequential order, it is sufficient to say that the commercial production of chilled orange juice began in Florida in large volume after World War II.

In order to maintain a state of freshness in chilled citrus juices similar to fresh juice or frozen concentrate, it is important to recognize that certain processing methods require special attention if the quality of the product is to be preserved. The quality of extracted citrus juices deteriorates rapidly, even under refrigeration, but the rates of change are reduced at low temperatures. It should be emphasized that extracted citrus juices are composed of the liquid phase of the cells of the juice vesicles as well as the soluble and insoluble cell constituents. Consequently, extracted juice is not, from an energy relationship, in equilibrium with its environment. In other words, it is physically and chemically unstable. This chemical instability is highly correlated with the losses of flavor and other quality characteristics of the juice which are caused chiefly by chemical changes and decomposition of its constituents, enzyme reactions, and microbiological activities (chiefly fermentation). In the production of frozen citrus concentrates, these deteriorative changes may be prevented by heating the juice before it goes to the concentrator. This same heating procedure is applicable for stabilizing citrus juice before rapid cooling and subsequent refrigeration. Heat treatment forms the basis for extending the production and distribution of chilled orange juice by stabilizing the juice and maintaining the fresh quality of the product during a period of time sufficient for large-scale marketing throughout the United States. After stabilizing the fresh juice by passing it through the desired heat exchanger, at controlled time and temperature, to avoid changes in flavor caused by excessive heat, the juice is rapidly cooled to 32° F. (zero centigrade) or lower, and maintained at this temperature until it is used by the consumer.

In addition to enzyme reactions and other chemical changes that occur in fresh juice, microbiological reactions, such as yeast fermentation, and other bacterial changes (*Lactobacillus, Leuconostoc mesenteroides,* and *Aerobacter*), must also be prevented in order to produce chilled citrus juices of good quality. Bacterial activity in relation to maintaining excellent physical and chemical characteristics of freshly extracted citrus juices has been discussed in considerable detail in chapter 4.

The demethylation of pectic substances in the juice by the naturally occurring pectinesterase produces clarification in the juice by separating or coagulating the suspended solids. The coagulum is the insoluble calcium pectate that is formed as a result of the reaction of the natural calcium ion with pectic acid, the demethylated product caused by the pectinesterase enzyme. Sedimentation of suspended solids proceeds at a slow rate in citrus juices when maintained at low temperatures. Pasteurization temperatures not only destroy microorganisms, but also inactivate enzymes, including pectinesterase, which are chiefly responsible for flocculation. It is important to recognize that the heat treatments used in pasteurizing fresh extracted citrus juices should be designed for the purpose of reducing microorganisms and inactivating the necessary enzymes without adversely affecting the taste and quality as well as the nutritional value of the juice. Chilled citrus juices are similar to frozen concentrates in that both microorganisms and enzymes produce deteriorating effects on their quality.

In order to supply the demand for chilled citrus products throughout the year, it is important to have access to a reserve supply of frozen concentrate for conversion to chilled citrus juice when the supply of fresh fruit is limited, as it is in Florida, from July through October. The flavor of the chilled juice depends on the quality of the fruit and the frozen concentrate used.

Juice Processing

CANNED GRAPEFRUIT JUICE, pasteurized and single-strength, was one of the earlier citrus-juice products to be placed on the market. Until 1943, grapefruit juice was packed in the largest amounts. Through research and development, the technology of canning finally advanced to a stage where enough scientific data had accumulated to permit the successful canning of orange juice. Blended orange and grapefruit juices are now canned in relatively large volume, with specifications as noted under "Blended Citrus Juices" in a preceding section of this chapter.

Riester *et al.* (1945) stated that progress in the improvement of quality of canned juice had been accomplished by recognition of the fundamental principles given here.

1. The use of good fruit at its optimum maturity.

2. Extraction of the juice in such a way that it will contain a minimum amount of naringin and proper amount of peel oil.

3. Rapid handling, from extraction through pasteurization, filling, and final cooling, is essential to minimize enzyme action and oxidation.

4. The application of a "high-short" pasteurization that is sufficient not only to prevent microbiological spoilage, but also to inactivate the pectic enzymes that are capable of destroying the colloidal suspension of the juice solids in the canned product.

All fruits for juicing purposes must have a ratio of at least 7 parts of soluble solids to each part of acid, calculated as citric. Well-matured grapefruit produces a processed juice of better flavor than that of less-mature fruit; the percentage of the bitter principle, naringin, is smaller; the segments do not break so easily; and a smaller amount of added sugar is needed to secure the desired sweetness.

Methods. Stevenson (1934), one of the earlier workers in the processing of grapefruit juice, pointed out that canned grapefruit juice, as then placed on the market, contained some suspended pulp in addition. The unsweetened juice was too sour for the average consumer, so that most of the commercial product was slightly sweetened by the addition of dry sugar or sugar syrup. In the latter case, the federal food and drug authorities ruled that the added syrup would have to contain at least 65 per cent sugar in order that dilution with excessive quantities of water might be avoided. Stevenson also reported that in extracting the juice, it was desirable to use methods which retained only a small amount of volatile peel oil. The presence in the juice of small amounts of peel was noted to be desirable because it added materially to both aroma and flavor. Burdick and Allen (1948) stated that 0.003 to 0.005 per cent recoverable oil appeared to be best for grapefruit juice.

After the juice has been extracted from the fruit, it is passed through a fine-mesh screen—the finishing unit. The screen most commonly used consists of a thin metal cylinder or cone made of monel metal or stainless steel, having small perforations. The size of the perforations determines to a considerable degree the amount of pulp left in the juice. There is a revolving beater inside the cylinder. The extracted juice is fed into the screen, which not only removes seeds and coarse fiber, but also breaks up the pulp into fine particles so that there is less tendency for it to separate in the can. The pulp content is significantly associated with the taste of the juice, as shown by the fact that those juices which contain less than 5 or 6 per cent pulp have a thin, weak taste, while those with more than 8 or 9 per cent pulp taste "thick," and often have a bitter flavor. It is important that the freshly extracted grapefruit juice move through the finishing unit as quickly as possible, thus avoiding the dissolution of excessive amounts of naringin from the rag portion (segment membranes) of the pulp. To avoid the appearance of excessive amounts of cellular materials, such as pectic substances, naringin, and/or limonin, in the juice, it should be efficiently screened by passing it through a series of rotating cylinders, operating according to definite specifications with respect to size of perforations and linear dimensions of the cylinders.

The juice is collected in suitable tanks where dry sugar or syrup of 65° Brix is added in sufficient quantity to give a concentration of 14 to 16° Brix. From this point, the juice may be handled in a number of ways, as shown on the following page.

1. It may be heated in the tank to a temperature of 160 to 180° F. (71 to 82° C.) and filled into the cans, which are then closed and pasteurized.
2. The juice may be filled into the cans cold; they are then passed through a hot-water exhaust box, closed, and pasteurized.
3. The juice may be filled into the cans cold; they are then closed on a vacuum closing machine and are finally pasteurized.
4. The cold juice may be subjected to flash pasteurization.

Excess head space in the cans results in abnormal internal corrosion and objectionable changes in flavor of the juice. For this reason, the cans are filled as full as possible at the time of closing. The heat treatment used after closing them naturally depends on the preceding treatment, but should in any case be sufficient to bring the center of the can to a temperature of at least 170° F. (77° C.). Whatever treatment is used, it should be followed by prompt and thorough cooling of the cans after the heat treatment is completed.

At the time Stevenson (1934) published his article, there were no government or trade standards for canned grapefruit juice, and there was no agreement as to what method of preparation and canning gave the best product. However, it was generally considered that prompt canning of the juice after extraction was essential to give a satisfactory product.

Shearon and Burdick (1948) pointed out that all equipment used in producing the juice would have to be stainless steel, monel metal, or aluminum because ascorbic acid would be catalytically oxidized by even small traces of copper or iron.

For further details on commercial processing methods, see the references cited at the beginning of this chapter.

De-aerating and De-oiling. According to Pulley and von Loesecke (1939), de-aeration, de-oiling, pasteurization, and inactivation of enzymes had been found to constitute the most important operations in canning grapefruit juice. They reported that the freshly extracted juice contained between 2 and 4 per cent of gases by volume. At least, most of the oxygen would have to be removed from the juice because of its adverse effects on color, vitamin C, and oils. The removal of dissolved oxygen would also prevent oxidative and flavor changes in the finished juice.

These gases are known to be easily removed by subjecting the juice in thin films to a high vacuum. As cited earlier, Burdick and Allen (1948) stated that for optimum flavor and storage life, the juice should contain between 0.003 and 0.005 per cent recoverable oil. De-aeration and the de-oiling evaporation are generally done continuously in a vacuum of 26 inches (66 centimeters) or higher. The oxygen and oil contents of grapefruit juice can be reduced to desirable levels by flash evaporation of the juice up to about 5 per cent of its volume.

Mottern and von Loesecke (1933) were among the first to use de-aeration and pasteurization to increase the keeping time of canned orange and grape-

fruit juices. Under such treatment, they were able to keep the canned juices for at least 4 months when stored at 35° F. (1.7° C.).

Filtration. Chace (1920) did some early work on the filtration of grapefruit juice. His method was to pasteurize the juice at 85° C. (185° F.), let the sediment fall, then filter through kieselguhr (infusorial earth).

Caldwell (1922) tried gelatin, casein, egg albumen, carbon, and other substances which were satisfactory for filtering some juices, but were not satisfactory for filtering grapefruit juice. Grapefruit juice, like all citrus juices, is especially difficult to filter because of its colloidal content. As indicated elsewhere, it is important to retain the cloud in grapefruit juice. Naringin is controlled by maturity of the fruit and by extraction methods, so that filtration is not necessary to reduce bitterness.

Joslyn and Sedky (1940) found that clearing of juices occurred at lower rates the higher the temperature or the longer the time of heating.

Pasteurizing Temperatures and Inactivation of Enzymes. Citrus juices are heated specifically to inactivate the pectic enzymes, so as to prevent the conversion of pectin to pectinic acids which will afterward coagulate, leaving a clear, supernatant liquid; secondly, to reduce and eliminate viable spoilage microorganisms. Since citrus juices are highly sensitive to heat, the time–temperature variable must be accurately controlled if the juices are to be pasteurized with a minimum of undesirable physical and chemical defects.

Nolte and von Loesecke (1940*c*) found that temperatures of 85 to 88° C. (185 to 190° F.) inactivated enzymes and subsequently prevented settling and curdling of the juice. "High-short" pasteurization was found to destroy all organisms that might otherwise have caused spoilage during storage or transportation.

Joslyn and Sedky (1940) also found that the enzymes in oranges and grapefruit were somewhat more heat-resistant than those in lemons, and that heat inactivation was more rapid at pH 2.5 than at pH 4.0 (see table 76, chapter 2). For centrifuged grapefruit juice, they found that lowering its pH decreased the time necessary to inactivate the enzymes at a given temperature. Stevens *et al.* (1950) obtained similar results. They found that grapefruit juice with a pH of 3.2 could be pasteurized in 2 minutes at a temperature of 84° C. (183° F.) and in 15 seconds at a temperature of 89° C. (192° F.). Rouse and Atkins (1952) reported that Duncan grapefruit juice was completely inactivated by heating for 0.8 second at temperatures of 195, 200, and 205° F. (90, 93, and 96° C.) when the pH of the juices was 3.0, 3.5, and 3.8 respectively. These data show that pasteurization of grapefruit juice is a time–temperature reaction, which indicates that longer times are required for pasteurization at lower temperatures, and conversely shorter ones at higher temperatures.

Although there are many variations in the methods and types of machinery that are used by citrus-juice processors, it is important to recognize that cer-

tain fundamental steps are the same for all operators who produce juice from the fresh fruit. These essential steps are enumerated here.

1. After receiving and storing the fruit in the plant, the sequence of operations includes washing and cleaning it in the presence of germicidal and detergent preparations, followed by grading and sorting. Preceding juice extraction, the fruit (especially grapefruit) must be sorted according to size, and this is accomplished by commercial sizers which can be purchased from an equipment supplier.

2. The fruit is conveyed to extractors which remove the juice and separate the peel for further processing into by-products. The extracted juice contains seeds and coarse pulp materials which may be removed by passing the juice through a finishing unit. Although the methods for screening citrus juices vary considerably in technical design, the screw-type finisher is representative of the kind of equipment ordinarily used to screen citrus juices. It consists chiefly of a helical screw enclosed in a cylindrical screen with .020- to .045-inch perforations.

3. The finished juice is pumped into blending tanks, where its quality is assessed in terms of total soluble-solids and acid contents.

4. After blending, the juice is de-aerated and de-oiled, for reasons that have been elaborated earlier in this chapter.

5. Pasteurization of single-strength citrus juices is performed, not only for the destruction of spoilage microorganisms, but for the inactivation of the enzyme pectinesterase, the activity of which is highly associated with cloud loss of the juice. The flavor of pasteurized canned juice differs from that of fresh juice, and changes in flavor continue to increase with changes in time and temperature of storage.

6. The hot pasteurized juice is conveyed immediately to the can-filling and closing equipment, after which the closed cans are rapidly cooled by rotation under sprays of cold water. A jet of steam is frequently introduced beneath the can lid before sealing, for the purpose of cleaning the inside of the lid as well as producing a vacuum within the can.

Pasteurized Concentrates. Wharton (1953) stated that in 1869, Welch first discovered a method for preserving unfermented grape juice. His discovery pioneered the entire industry of canned and bottled fruit and vegetable juices. McDermott (1917) was one of the first to salvage grapefruit culls. He began his work in 1911. He extracted the juice from culls, pasteurized it, and was able to keep it for 18 months. He also concentrated grapefruit juice to 20 per cent of its original volume by vacuum evaporation. However, his work was not done on a commercial basis.

Concentrated pasteurized citrus juice was produced and used commercially for beverage bases, sherbets, and candies long before frozen concentrates appeared on the market. This product had limited use as a reconstituted single-strength juice because it developed off-flavors during processing and deterioriated rapidly in storage. Since these concentrates are generally intended for storage above the freezing point (between 35 and 40° F.—1.7 and 4.4° C.), it is highly important that the juice entering the evaporator be in a sterile condition. Flash pasteurization inactivates the pectic enzymes to prevent cloud loss, and destroys the spoilage microorganisms. After the juice is

concentrated, it is usually reheated to approximately 170° F. (77° C.), poured into cans while hot, and hermetically sealed and cooled. In a large-scale operation, the juices are successfully concentrated with the low-temperature, falling-film type of evaporator, which operates in a temperature range of 60 to 80° F. (16 to 27° C.). This low-temperature type results in a product of higher quality than the types that operate at higher temperatures, between 100 and 120° F. (38 and 49° C.). As the technical efficiency of the low-temperature, high-vacuum evaporators has increased, the older types of evaporators have continued to be replaced by the most modern equipment.

High-density citrus juices are produced in a wide range of concentrations, depending on the required specifications. High-density orange juice that is packed in 1-gallon (3.79-liter) cans or paraffined barrels has a concentration between 60 and 70° Brix. Grapefruit juice is usually concentrated to a lower Brix value than orange juice. As a result of certain conditions in the older types of evaporators, pasteurized grapefruit concentrate was found to turn brown during storage, the degree of browning being in direct relationship to the degree of concentration.

According to the data of Curl *et al.* (1946), concentrated orange juice at 65° Brix should be stored at 40° F. (4.4° C.), where only slight physical and chemical changes would occur. In fact, no significant browning was observed in concentrates held in storage for 1 year at that temperature. At 80° F. (27° C.) and above, the color of the concentrate turned brown, and the flavor became "ordinary to poor" in 6 months. After 3 months' storage at 95° F. (35° C.), the color became brown, and the flavor developed from "poor" to "very poor." After 1 month's storage at 120° F. (49° C.), the samples turned dark brown and developed a "very poor" flavor.

Gas formation in canned citrus concentrates is a type of spoilage that develops swells in the can, by either chemical decomposition or yeast fermentation. The relationship between storage time and temperature to gas formation in canned orange concentrates was experimentally demonstrated by the fact that swells developed in cans within 2 weeks at 120° F., 90 days at 95° F., and 6 months at 80° F. (Curl *et al.*, 1946).

Carbon dioxide proved to be the principal gaseous constituent that produced the swells in cans caused by either fermentation or chemical decomposition. Curl (1948) reported that all samples of orange concentrate containing 41 per cent or more of soluble solids produced gas after storage for 60 days at 120° F. Gas production was markedly accelerated by adding ascorbic acid to the samples. Synthetic mixtures containing sugars, ascorbic acid, and amino acids evolved gas in amounts equivalent to those produced in concentrated orange juice. Samples of concentrate from cans that produced swells as a result of fermentation retained satisfactory color and flavor, with only slight changes in chemical composition, but samples from cans in which swells

were attributed to chemical decomposition showed considerable darkening and deterioration in flavor.

Citrus Beverage Bases. Citrus-juice concentrates, the essential oils, and citric acid are the important constituents used in compounding beverage bases. These bases, produced from citrus-juice concentrates, are manufactured by fruit processors, and are sold to the retail trade, where they are mixed with sugar and water to produce the finished beverages. Orange-juice beverages are produced in higher commercial quantities than any other citrus-fruit drinks.

Bottler's base for carbonated drinks is usually prepared by concentrating pasteurized citrus juice to 65° Brix under vacuum. The juice should be pasteurized at 200° F. (93° C.) for about 4 seconds before concentrating, to inactivate the pectic enzymes and thus stabilize the cloud so that the final carbonated drink will have a turbid, fruitlike appearance. The concentrate is mixed, under refrigerated conditions, with calculated amounts of sugar, citric acid, citrus oil, and food coloring, plus benzoate of soda as a preservative. The mixture is homogenized, pasteurized again at 170° F. (77° C.), filled hot into 1-gallon enamel cans, and sealed. Carbonated citrus beverages are usually composed of approximately 6 per cent fruit juice (USDA, 1956). Although grapefruit juice has an excellent flavor, the volume used in beverages has never equaled the quantity used from oranges, lemons, and limes. Grapefruit base is produced in limited amounts for the dairy industry, and the beverage produced contains approximately 15 per cent juice. Carbonated grapefruit drinks are commercially produced, and sold as mixers. It should be noted that citrus beverages are usually poor sources of vitamin C as compared with reconstituted frozen concentrate or canned single-strength juice. Beverages lose vitamin C at a rapid rate, the losses amounting to as much as 70 per cent in a week's time, depending on the product (Roberts, 1937).

Frozen Grapefruit Concentrate. This product is produced with essentially the same equipment that is used to produce frozen orange concentrate. Grapefruit juice that is converted to frozen concentrate has to be heated from 150 to 180° F. (66 to 82° C.) to prevent gelation and clarification during storage. Since grapefruit juice contains more pectin and pectic enzymes than orange juice, it is also more susceptible to gelation than orange juice. This thermal stabilization process usually takes place before the juice goes to the evaporator.

The basic principles involved in the preparation of high-quality frozen citrus concentrates are concerned with the process of evaporating the juice under high vacuum at low temperatures. Since the boiling point of a liquid is defined as the temperature at which its vapor pressure is equal to the atmospheric pressure, it follows that the boiling temperature of a liquid can be markedly reduced by heating it under greatly reduced atmospheric pres-

sure. Consequently, when citrus juices are evaporated at 80° F. (27° C.) or below, the product developed has no cooked or off-flavor, but the concentrate has a flat taste, and the fresh-juice aroma is not present, because most of the volatile flavoring constituents have been distilled with the water vapor. These volatile aromatic constituents, lost during evaporation, can be returned to the concentrate (60 per cent soluble solids) by adding freshly prepared single-strength juice in sufficient amounts to reduce the concentration to 42 to 45 per cent soluble solids. The fresh juice, the so-called "cut-back" juice, restores fresh aroma and flavor to the concentrate. The cut-back juice, which contains a comparatively large quantity of suspended solids, gives the concentrate a fresh-juice appearance for consumer use on dilution to normal strength.

After the cut-back juice has been added to the concentrate, the latter is transferred to cooling tanks which are maintained at –10° F. (–23° C.). In these coolers, the concentrate is quickly cooled to 40° F. (4.4° C.) or below. This chilled 42° Brix concentrate is moved to the filling equipment, where it is run into cans, lidded, and slowly moved by conveyors through the freezers at –20° F. (–29° C.). While in the freezer, the contents of the cans are frozen, and they are then cased for shipment by refrigerated transport or placed in storage at –10° F.

Methods. The types of low-temperature evaporators that have been used for the concentration of citrus juices have passed through many modifications and variations in design. Multiple-, double-, and triple-effect evaporators are generally employed where emphasis is placed on the comparatively short exposure of the juice to heat, and where the concentration of the juice must be performed at reduced atmospheric pressures if the original qualities of the fresh juice are to be preserved and maintained.

During the many years of research and development of evaporating equipment for concentrating citrus juices, 3 distinct types of low-temperature evaporators have been used in Florida at various times. They are known as single-effect single-pass, single-effect with recirculation, and multiple-effect with recirculation (Schwarz, 1951). In these various evaporator types, the water-vapor pumping system is usually composed of a steam recompression unit or a refrigeration cycle.

The evaporation of a volatile liquid is dependent on 2 essential factors: (1) the supply of necessary heat of vaporization to the liquid, and (2) the importance of removing the evolved vapor so as to prevent its accumulation at the surface of the liquid. Consequently, the heat required for evaporation is almost entirely converted into latent heat of vaporization, and is carried out of the evaporator in this form by the vapor, to be utilized again by condensing the vapor. In evaporating water at atmospheric pressure from an aqueous solution, the heat consumed (chiefly latent heat) is in the water

vapor. It cannot be employed to evaporate more water under atmospheric conditions because, when the vapor is condensed, the liberated heat that was evolved at the boiling point of water (100° C.; 212° F.) produces no temperature difference to force the heat through the necessary heating surface into the fresh solution to be evaporated. On the other hand, if a liquid such as alcohol with a boiling point lower than that of water is distilled, the water vapor from the evaporator can readily be used as a heat source by condensation in the heating coils of the alcohol still. Continuing this process, if another liquid such as ethyl ether with a boiling point lower than alcohol is distilled, the heating coils of the ether still can serve as a condenser for the alcohol vapor, thus avoiding the use of boiler steam to distill the ether. In this system, a series of liquids with progressively lower boiling points can be evaporated with a single supply of heat.

The foregoing information illustrates the multiple-effect principle, which can be defined in terms of a process where a given quantity of heat can be used a number of times for evaporating a series of liquids with successively lower boiling points. Since the boiling point of an aqueous solution is a function of the pressure above the liquid, citrus juices can be concentrated at temperatures similar to those applied to the organic liquids of low boiling point just mentioned, provided the process is performed at the desired reduced pressure. In concentrating citrus juices in a vacuum, the principal advantages derived are: (1) reduction of the boiling point of the juice, (2) acceleration of its evaporation as a result of the increase in temperature difference between the boiling point of the juice and the low-pressure heat source, and (3) citrus juices deteriorate and develop off-flavors when they are boiled at normal atmospheric pressure.

Some Physical and Chemical Properties of Frozen Grapefruit Concentrates

Pectinesterase activity was studied by Rouse *et al.* (1959) as it affected the stability of frozen grapefruit concentrates sampled from various commercial stocks during the 1955–56 and 1956–57 seasons. Their general conclusions follow. The physical stability of citrus concentrates or reconstituted juices is dependent upon maintaining their cloud or turbidity. The suspended and soluble pectic substances are responsible for the cloudy and turbid characteristics of citrus juices, and the enzymatic breakdown of these materials by pectinesterases results in clarification or loss of turbidity. Although heat treatments inactivate pectinesterases and enhance cloud stability, the pectinesterase activity is not perfectly correlated with increased stability.

The frequency distribution of the pectinesterase activities shows that 89.3 and 94.8 per cent of the samples, respectively, for both seasons contained

TABLE 190

FREQUENCY DISTRIBUTION OF PECTINESTERASE ACTIVITY, INITIAL PULP CONTENT, AND PECTIN CONTENT IN RECONSTITUTED JUICES FROM FROZEN CONCENTRATED GRAPEFRUIT JUICES FOR THE 1955–56 AND 1956–57 SEASONS.*

PECTINESTERASE ACTIVITY		
Pectinesterase activity	28 samples 1955–56 survey (per cent of total)	96 samples 1956–57 survey (per cent of total)
0.0– 0.9	53.6	21.9
1.0– 1.9	25.0	53.1
2.0– 2.9	10.7	19.8
3.0– 3.9	7.1	5.2
4.0– 4.9	3.6	0.0

INITIAL PULP CONTENT		
Pulp by volume (per cent)	28 samples† 1955–56 survey (per cent of total)	96 samples‡ 1956–57 survey (per cent of total)
3.0– 3.9	3.6	0.0
4.0– 4.9	21.4	1.0
5.0– 5.9	32.1	1.0
6.0– 6.9	10.7	10.5
7.0– 7.9	17.9	25.0
8.0– 8.9	7.1	29.2
9.0– 9.9	0.0	15.6
10.0–10.9	3.6	12.5
11.0–11.9	3.6	3.2
12.0–12.9	0.0	1.0
13.0–13.9	0.0	0.0
14.0–14.9	0.0	0.0
15.0–15.9	0.0	1.0

PECTIN CONTENT		
Pectin, water-soluble (mg/100 g serum)§	28 samples 1955–56 survey (per cent of total)	96 samples 1956–57 survey (per cent of total)
15–19	7.1	0.0
20–24	14.3	9.4
25–29	28.6	14.6
30–34	28.6	20.8
35–39	14.3	25.0
40–44	0.0	17.7
45–49	7.1	9.4
50–54	0.0	1.1
55–59	0.0	0.0
60–64	0.0	1.0
65–69	0.0	1.0

*From Rouse *et al.* (1959).
†International Centrifuge, Size 1, Type SB, 1,700 rpm, 15 minutes.
‡International Centrifuge, Size 1, Type SB, 1,300 rpm, 10 minutes.
§Centrifuged juice was called serum after separation of pulp content.

fewer than 3.0 pectinesterase units (table 190). These low values had a tendency to increase the total storage period of the concentrates by increasing their physical stability or cloud retention. The pulp content was a measure of the "free and suspended pulp," calculated as percentage of pulp by volume in the reconstituted juices. In samples for the 1955–56 season, 92.8 per cent of the juices contained from 3 to 10 per cent pulp; in the 1956–57 samples, only 82.3 per cent of the juices were within this range. Although the juice samples for the 2 seasons were centrifuged at different rates (1,700 rpm for 15 minutes in 1955–56; 1,300 rpm for 10 minutes in 1956–57), the water-soluble pectins were not affected by this variation in centrifugal rate in determining the pulp content. Under these experimental conditions, approximately 18 per cent of the 1956–57 samples contained more than 10 per cent pulp by volume—the maximum amount permitted by United States standards for a grade A product.

The water-soluble pectins were determined on the centrifuged juices which had been previously reconstructed from frozen concentrated grapefruit juices (table 190). In the 1955–56 season, 92.9 per cent of the samples varied from 15 to 40 milligrams of water-soluble pectic substances and, in the 1956–57 season, 69.8 per cent of the samples fell within this concentration interval, while the remainder had a soluble-pectin concentration ranging from 40 to 67 milligrams per 100 grams of serum. The average amounts of soluble pectic substances in the juice samples for the 2 experimental seasons consisted of 30 and 35 milligrams per 100 grams of serum, respectively.

The frequency distributions of the water-insoluble and alcohol-insoluble solids of the juice samples are recorded in table 191. The data show that 89.2 and 87.5 per cent of the juice samples for the 2 seasons contained water-insoluble solids in amounts less than 99 milligrams per 100 grams of juice. The alcohol-insoluble solids consisted of not only the "free" and suspended solids, but pectic substances, proteins, and other hydrosols of high molecular weight, which were flocculated by the addition of 66 per cent isopropyl or ethyl alcohol, and were miscible in the water, serving to dehydrate the colloid particles. As would be expected, the values for alcohol-insoluble solids were much greater than those recorded for water-insoluble solids. The minimum values for the 2 seasons were 146 and 168 milligrams per 100 grams of reconstituted grapefruit juice, respectively; the maximum values for the same data amounted to 424 and 590 milligrams per 100 grams.

The rate at which clarification occurs in citrus-juice concentrate is a measure of its stability with reference to enzymatic reactions that may take place in transportation or storage.

The amount of light that it is theoretically possible to transmit through centrifuged citrus juices under specified conditions is highly associated with the degree of clarification.

TABLE 191

FREQUENCY DISTRIBUTION OF WATER-INSOLUBLE SOLIDS, ALCOHOL-INSOLUBLE SOLIDS, AND LIGHT TRANSMITTANCE IN RECONSTITUTED JUICES FROM FROZEN CONCENTRATED GRAPEFRUIT JUICES FOR THE 1955–56 AND 1956–57 SEASONS.*

WATER-INSOLUBLE SOLIDS

Water-insoluble solids (mg/100 g juice)	28 samples 1955–56 survey (per cent of total)	96 samples 1956–57 survey (per cent of total)
25– 49	14.2	5.2
50– 74	50.0	55.2
75– 99	25.0	27.1
100–124	3.6	9.4
125–149	3.6	3.1
150–175	3.6	0.0

ALCOHOL-INSOLUBLE SOLIDS

Alcohol-insoluble solids (mg/100 g juice)	28 samples 1955–56 survey (per cent of total)	96 samples 1956–57 survey (per cent of total)
100–149	3.6	0.0
150–199	17.8	10.4
200–249	42.8	29.2
250–299	14.3	22.9
300–349	17.9	18.8
350–399	0.0	2.1
400–449	3.6	6.2
450–499	0.0	4.2
500–549	0.0	0.0
550–599	0.0	6.2

LIGHT TRANSMITTANCE

Light transmittance† (per cent)	28 samples 1955–56 survey (per cent of total)	96 samples 1956–57 survey (per cent of total)
35.0–39.9	7.1	4.2
40.0–44.9	7.1	39.6
45.0–49.9	57.2	43.7
50.0–54.9	28.6	10.4
55.0–55.9	0.0	2.1

*From Rouse *et al.* (1959).
†Cloud measured as percentage of light transmittance of centrifuged reconstituted juice, using Lumetron 402-E Colorimeter.

As a result of the 2-year sampling period, 71.4 and 87.5 per cent of the grapefruit samples for 1955–56 and 1956–57, respectively, fell within a range of 35 to 50 per cent light transmittance, which was well within the arbitrary range of 0 to 69 per cent light transmittance, indicating no clarification for these samples (table 191).

Rouse *et al.* (1959) performed further experiments on the stability of grapefruit concentrates by measuring their rates of clarification and gelation. Grapefruit concentrates from the 1955–56 survey were thawed and stored for 48 hours at 40° F. (4.4° C.); the samples from the 1956–57 survey were thawed and stored for 96 hours, also at 40° F. In the 2-year survey, clarification occurred to some degree in only 3 per cent of the 1956–57 samples, indicating the effect of the extended storage period on the stability of the concentrates. With reference to the intensity of gelation in the samples, 82 and 92 per cent of the concentrates for 1955–56 and 1956–57, respectively, were free of gels. The remaining percentages of the samples for both seasons showed only slight gelation. None of the concentrates produced semisolid or solid gels.

Viscosity of Commercial Frozen Concentrated Grapefruit Juice. Ezell (1959) reported some interesting relationships in the viscosity data of orange and grapefruit concentrates. The results obtained from 27 samples collected from 10 Florida plants showed that the viscosity values of grapefruit concentrates, as measured with the LVF Viscometer, varied from 150 to 1,885 centipoises, in which 93 per cent of the samples fell below 1,000 cp and 67 per cent were below 500 cp.

In the absence of certain physical and chemical data that would affect the viscosity values of grapefruit concentrates, it was thought appropriate to present graphs that showed the effects of concentration (Brix) and temperature on the viscosity of orange concentrates, with the resultant effect that these relationships would probably be applicable to grapefruit concentrates. The viscosity of "true liquids" decreases about 2 per cent for each degree of rise in temperature. Consequently, temperature control is highly important in determining the apparent viscosities of frozen orange concentrates (42° Brix).

The viscosity of a solvent is generally increased by dissolving large quantities of solids in the solvent. Suspended particles can also cause increases in viscosity in proportion to the volume of suspended material relative to the total volume. The viscosity of hydrophilic colloids is generally high. In certain emulsoid sols, the viscosity increases to the extent that pressure is required to cause flow. In a biological liquid, such as orange juice, the apparent viscosity increases parabolically with the increase in concentration of soluble solids (degrees Brix), as well as with the amount of suspended pulp. This is satisfactorily demonstrated in the experimental data of figure 182, which

show that the concentration (degrees Brix) of a juice is a variable that must be reckoned with in comparing and evaluating the viscosity measurements of such systems.

The graph curve that shows the juice with 9.5 per cent pulp had lower viscosity values at every concentration level than the curves for those juices with pulp contents of 11.0, 13.0, and 15.5 per cent. This indicates that the apparent viscosity values increased very sharply with an increase in suspended pulp, as well as with an increase in soluble solids.

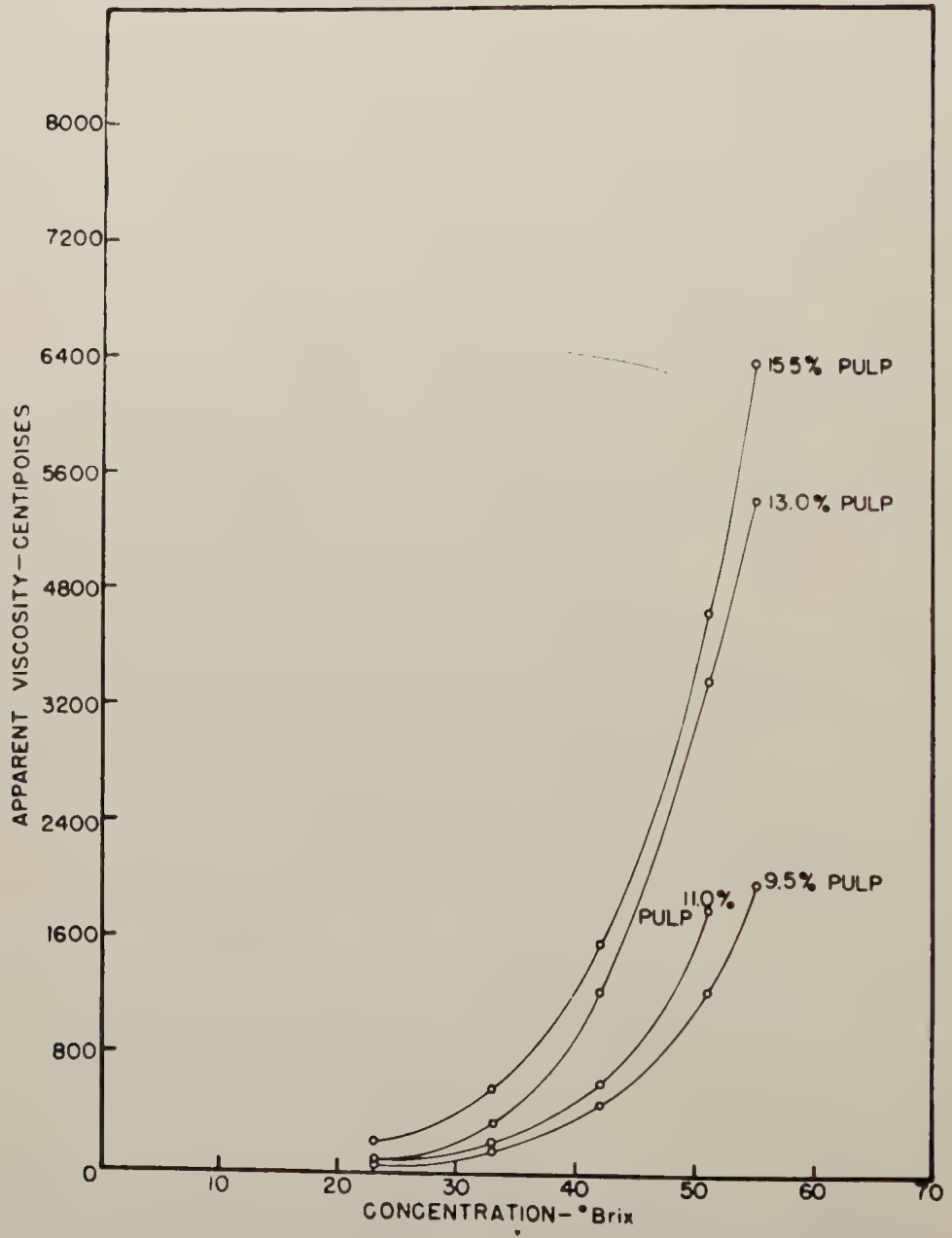

Fig. 182. Effects of concentration and pulp content on the viscosity of frozen concentrated orange juice. (From Ezell, 1959.)

The data in figure 183 show that the apparent viscosities of 4 commercial orange concentrates decreased logarithmically with increases in temperature. Furthermore, the viscosity of true liquids of homogeneous solution does not change with variations in rates of shear, but is determined by the concentration and temperature at which the measurements are made. In other words, the increased rate of flow (rate of shear), plotted against the increase in force applied (shearing force), is a linear function which passes through the point of origin. In contrast to the linear relationships of true viscous flow, colloidal materials with various degrees of hydration, when in suspension, change in resistance to flow with a change in rate of shear (non-Newtonian liquids).

Foam-Mat Drying Procedure for the Dehydration of Citrus Juices

CITRUS-JUICE CONCENTRATES and many liquid foods can be converted to stable foams and subsequently dried rapidly in air to produce instant powders without undue changes in quality. This method of dehydrating liquid foods is commonly referred to as the foam-mat drying procedure (Morgan *et al.*, 1961; Graham *et al.*, 1965). The rapid drying is the result of the movement of moisture by capillary activity from the liquid films surrounding the bubbles dispersed in the foam. Suitable foams are produced by the use of small quantities of edible stabilizers, and the most efficient stabilizers discovered are solubilized soya protein, albumin, sucrose fatty-acid esters, glyceryl monostearate

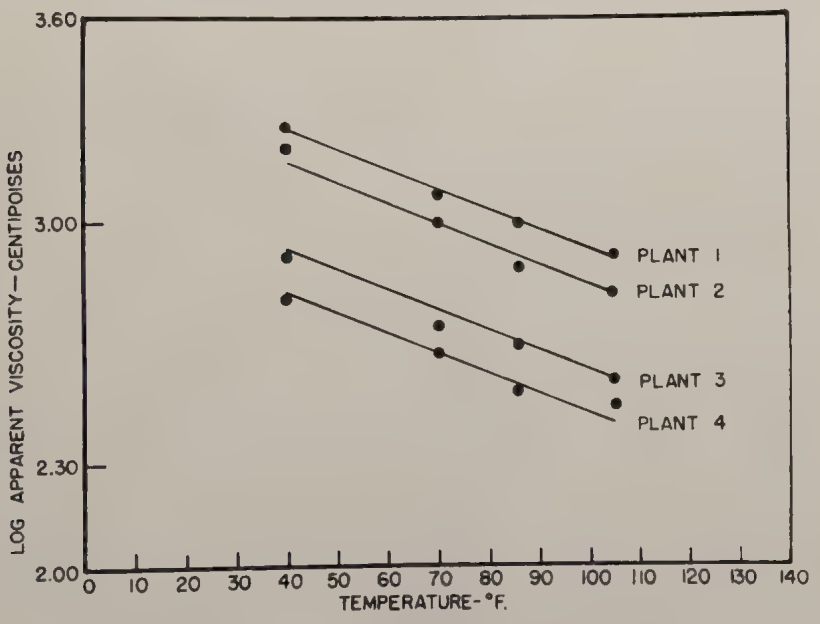

Fig. 183. Effects of temperature on the viscosity of 4 commercial samples of frozen concentrated orange juice. (From Ezell, 1959.)

(GMS), and methocel 10 cps (MC). Instant reconstitution of the powders results from retention of the honeycomb structure in the dry solid.

The object of this summary is to report chiefly on the principles involved in the foam-mat drying method and its application to the production of powders from grapefruit concentrates. Since methods for foaming of grapefruit concentrates were developed by using earlier experimental data with orange-juice concentrates, it was thought advisable to include in this review some related data on foam-mat dried orange concentrates.

Foams and Foam Characteristics. From the viewpoint of a liquid, foam is a physical system which is composed of a gas or gases dispersed in a liquid; i.e., the gas is the dispersed phase, and the liquid is the dispersion medium. The initial stage in the production of a foam is the formation of numerous gas bubbles in the interior of the liquid, and this condition is accomplished by means of agitation, but agitation is ineffective unless the chemical and physical condition of the liquid is suitable. In a pure liquid, agitation never produces small, stable bubbles, and the larger bubbles that are formed are continually uniting. Therefore, a pure liquid, whether its surface tension is high or low, does not aerate to any great extent; thus, water, with 3 times the surface tension of amyl alcohol, aerates to about the same degree as amyl alcohol. When the surface tension of a liquid is reduced by the addition of a solute, the solute becomes concentrated in the surface layer (interphase). The surface concentration is probably related to $-c(ds/dc)$, where c is the concentration in the bulk of the solution, and s is the surface tension of the solution. Therefore, when ds/dc is negative, a positive surface concentration occurs.

From a practical standpoint, the way to attain a large liquid surface is to form a multitude of bubbles; in other words, to form a foam. As just shown, a stable foam cannot be formed with pure water or with any other pure liquid, but when small amounts of certain substances are added to these systems, they take on the property of foaming when agitated in the presence of air or inert gases, such as nitrogen. The stability of the foam varies greatly with the substance added, and the substances that produce the most stable foams may also produce undesirable side reactions.

Foam-Mat Drying Process. The operating principles of the foam-mat drier are adequately illustrated by the flow diagram of figure 184 (Rockwell *et al.*, 1962). This process consists of drying thin layers of stabilized foam materials in heated air at atmospheric pressure and humidity. Liquid food or food concentrate is initially converted to foam by mixing in a closed container in the presence of an inert gas, and a small quantity of an edible foam stabilizer when it is required. The foam flows to a feeder (or foam spreader), whence it is spread in a thin layer on the perforated sheet-metal trays, and then craters are formed in the foam layers by blowing thin jets of air up through

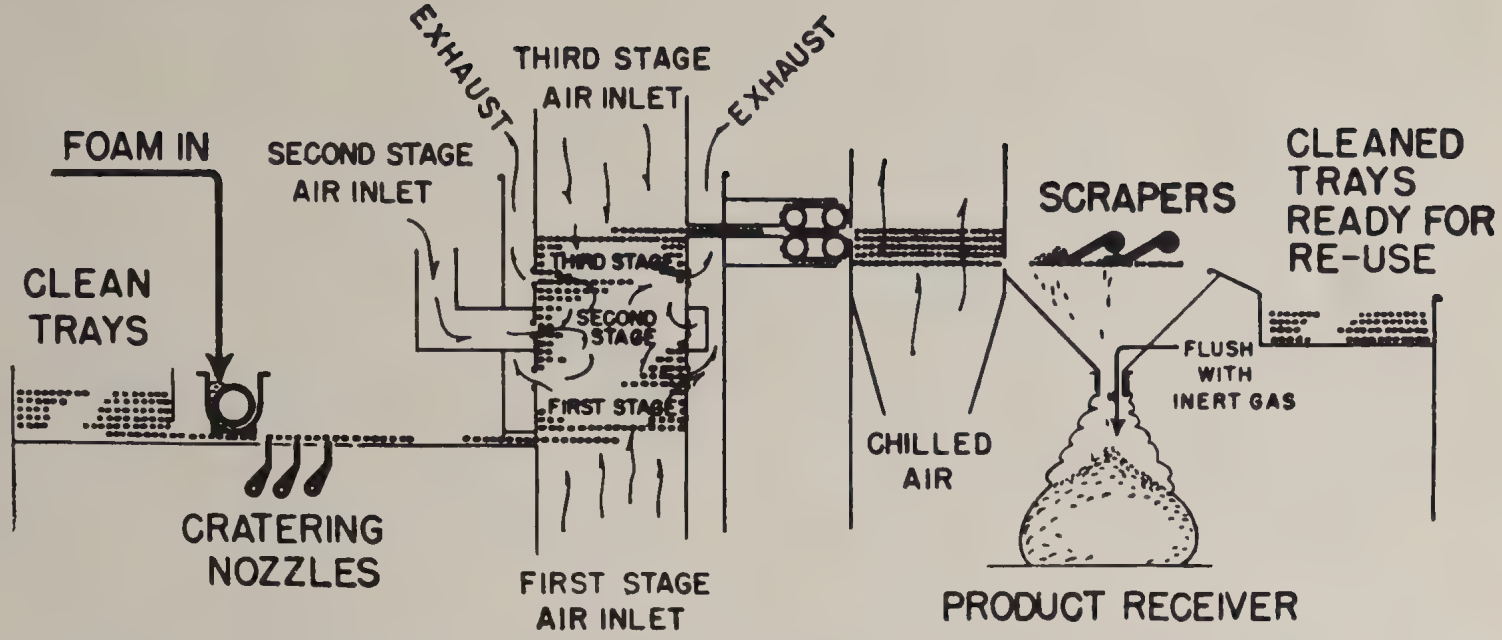

Fig. 184. Process flow diagram. Trays move from left to right under feeder and over cratering nozzles into bottom of drier stack. Trays of dry product move from top of drier to cooling stack, then are scraped for detraying. The product is flushed with inert gas in receiver. (From Rockwell *et al.*, 1962.)

the tray apertures. Freshly cratered foam is reduced in moisture content between 2 and 4 per cent in a single-stack, multiple-stage drier. The dried material is cooled, removed from the trays, and is finally packaged in a room with a relative humidity not exceeding 15 per cent. As a result of large surface areas, foams are dried at a rapid rate, and the dried product, which has an extremely porous structure, can be easily ground to a fine powder that can be instantly rehydrated in cold water. Foam-mat dried foods are usually superior in quality to the ordinary air-dried products.

Further comment should be made about the drying procedure in the drier stack (figure 184). The multiple-stage drying is designed to give a high-capacity concurrent stage for the wet foam, an intermediate stage for continued drying, and a countercurrent stage for the final drying. In the first stage, air at 220° F. (104° C.) enters at the bottom of the stack, and flows up at 400 feet per minute[2] through approximately 6 trays before being discharged through exhaust openings at 140° F. (60° C.) in an unsaturated state. In the second stage, air at 170° F. (77° C.) enters openings around the middle of the stack, and flows in both directions at about 150 fpm to exhaust ports between the first and third drying stages. In the third stage, air at 130° F. (54° C.) enters at the top of the stack, and flows down through the trays at about 50 fpm. The efficiency of this multiple-stage drying procedure is indicated by the fact that 80 per cent of the moisture is removed in the first stage, while the remaining 20 per cent is mostly removed in the second stage. Uniformity of the moisture content of the dried material is ascertained in the third stage.

Producing Grapefruit Crystals by the Foam-Mat Drier. Berry *et al.* (1967*a, b*) produced grapefruit powders from concentrated juice by a foam-mat procedure similar to the one just described. The maximum temperature of the product in the drier was 170° F. (77° C.) for a 2-minute exposure, and it reached 150 to 160° F. (66 to 71° C.) during the remainder of the drying time (10 minutes). After chilling the dried material, its final moisture content was 1 to 1.25 per cent, which was low enough to require no desiccant in the packaged product. Before packing, this was ground to a powder and afterward passed between pressure rolls, which served to increase its density to approximately 0.6 gram per cubic centimeter.

Berry *et al.* (1967*a, b*) used grapefruit concentrates of 40 to 58° Brix in producing grapefruit powders. Both nitrogen and carbon dioxide were used as foaming gases, being released into the mixing head at various pressures. The mixing head revolved at different rates of speed, and the back pressure was measured by a gauge located near the point where the concentrate entered it. The temperature of the foam was measured by thermocouples placed at the outlet of the mixing head. Foam characteristics were evaluated before drying by measuring the foam density in grams per cubic centimeter and by estimat-

[2] One cubic foot equals 28.3 cubic decimeters.

ing the bubble-size index. Typical foams had a density of about 0.30 gram per cubic centimeter and a bubble-size index of about 24 (average bubble diameter in microns). Grapefruit foams with these characteristics were found to be suitable for drying, as noted by Berry *et al.* (1965*b*).

The stabilizing agents used with grapefruit concentrate were methyl cellulose, modified soya albumin, glyceryl monostearate, various algins and pectins, and cellulose derivatives. Solutions or suspensions of these substances were fed into the mixing head of an 8-inch (20-centimeter) Oakes mixer at various rates.

The noncitrus additives should be limited to 1 per cent; at present, satisfactory foams are being produced with no more than 0.5 per cent additives.

In these experiments, the typical drying conditions for dehydrating grapefruit juice consisted of passing hot air at 254° F. (123° C.) at about 522 cfm through and around the trays of the first effect of the multiple drier. In the second effect (middle section), the air entered at 178° F. (81° C.) at 366 cfm. In the third effect (upper section), the air was 141° F. (61° C.), with less than 10 per cent relative humidity and at the rate of 555 cfm.

It is well known that many biological materials contain definite percentages of moisture when exposed indefinitely to air of a given temperature and humidity. This water is probably adsorbed; and, under the circumstances, it represents a true dynamic equilibrium. Furthermore, the moisture percentage equilibrium for a given percentage of relative humidity always decreases as the temperature increases. In foam-mat dried citrus powders with 1 per cent moisture, the relative humidity equilibrium was stated to be about 13 per cent at room temperature by Wagner (1961). It is apparent from these facts that relative humidity is highly important in removing the moisture from citrus juices. When ambient air at 90° F. (32° C.) and relative humidity of 70 per cent is heated to 140° F. (60° C.), the relative humidity is above 17 per cent. To reduce the relative humidity of the entering air during the warm, moist periods of the year, a refrigerated type of dehumidifier was installed on the third effect of the drier just described. This unit was used to reduce the moisture content of the ambient air when it had more than 10 per cent relative humidity.

Evaluation of Foams for Foam-Mat Drying. In the past, foams that met requirements for foam-mat drying were evaluated by foam density in grams per cubic centimeter, and it was observed that this value was only partially associated with the factors that influenced a foam's drying properties. Berry *et al.* (1965*a*) noted that earlier researchers had observed, from microscopic studies of foams, that bubble size was related to the ease of moisture removal.

As a result of greatly increased interfacial surface, the foams that contain the smaller bubbles are the ones that are dried more thoroughly under milder conditions. Conversely, the foams most suitable for drying are the ones with

the smallest bubble size. This relationship is the basis of the bubble-size test that can serve as a supplement to the density determination for the evaluation of foam characteristics that is so essential in the drying of citrus juices by the foam-mat method.

Without elaborating on the detailed technique of the "bubble-size" determination of foams, it is sufficient to say that microphotographs were taken of the foam samples, and bubble-size indices were calculated that proved to be roughly proportional to the mean bubble diameter. The bubble-size index can be ascertained, therefore, by dividing the area of the field of observation by the number of bubbles in the observed area, and the resultant quotient is the average area of the circle. The diameter of the circle of this area is designated as the "bubble-size index," and is approximately the mean bubble diameter in the foam (Berry *et al.*, 1965*a*). Because of the circular nature of the bubbles, the total area of observation is not completely occupied, and no correction is made for this unoccupied area in the calculation of the "bubble-size index." As the bubbles are reduced in size and become more uniform, the "bubble-size index" approaches the true mean bubble diameter. This index number has been reported to be significantly correlated with visual observation of bubble size and with the drying characteristics of citrus-juice foams.

The food materials that are dried by foam-mat procedures must have not only flowability and uniformity, but also a high degree of moisture release under drying conditions. The data in table 192 (Berry *et al.*, 1965*a*) show the relationship between the "bubble-size index" and the moisture contents of powders produced independently from both grapefruit and orange concentrates. The tabulated data also show that foams produced from 2 different ratios of MSA:MC had larger bubble-size indices than those prepared from GMS. The grapefruit and orange foams were dried at 180 and 170° F. (82 and 77° C.), respectively. The dehydrated product prepared from foams with

TABLE 192

RELATIONSHIP OF "BUBBLE-SIZE INDEX" OF FOAM TO MOISTURE LEVEL OF POWDERS PRODUCED.*

Fruits tested	Drying temperatures (F. and C.)	Foaming agents and ratios used (solids basis)†	Bubble-size index	Moisture (per cent)
Grapefruit	180 (82)	MSA:MC 7:1	39	2.60
		2:1	30	2.21
		GMS	13	0.89
Orange	170 (77)	MSA:MC 2.3:1	50	2.50
		3.8:1	28	1.86
		GMS	14	0.94

*From Berry *et al.* (1965*a*).
†Symbols: GMS is glyceryl monostearate; MC is methocel (10 cps); MSA is modified soya albumin.

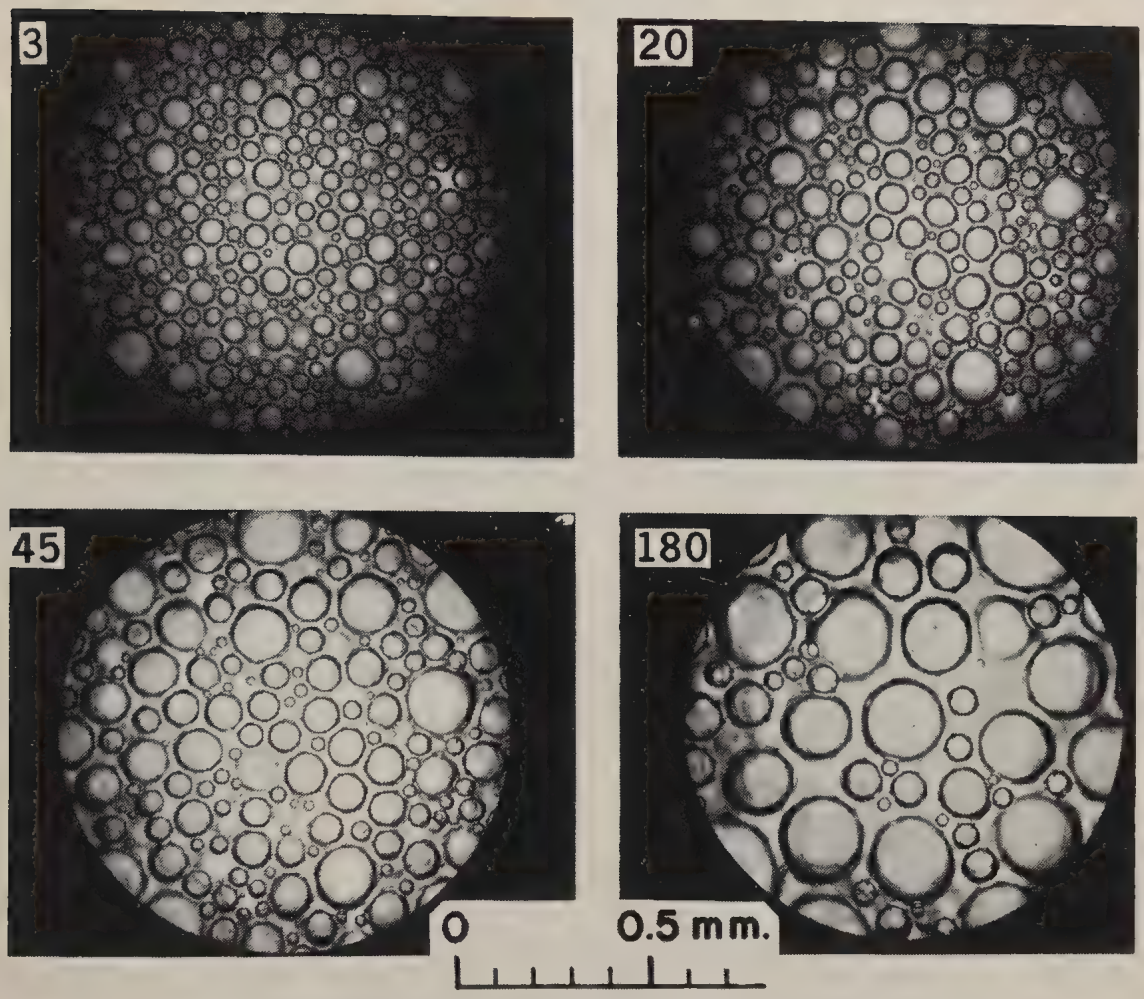

Fig. 185. The growth in average bubble size with time (shown in minutes in insets). (From LaBelle, 1966.)

smaller mean bubble diameters also had smaller percentages of moisture. This fact is definitely illustrated by the grapefruit data in the table, which show that foams with bubble-size indices of 39, 30, and 13 produced powders containing 2.60, 2.21, and 0.89 per cent moisture.

LaBelle (1966) subjected orange-juice foam to certain objective tests with respect to rapid changes in bubble-size diameter with time. The photographs in figure 185 show the growth in the average bubble size in orange foam with time. During the time intervals of 3, 20, 45, and 180 minutes, the mean bubble diameters (μ) increased to 54, 72, 95, and 163 μ, respectively. These values corresponded to a bubble count ($\Sigma n/mm^2$) of 321, 231, 162, and 58. These data seem to indicate that the bubble sizes increased in fresh foams immediately after formation. In estimating the bubble population in a given plane, the values were biased in the direction of large bubbles, since the depth of the field (15 to 25 μ) was considerably less than the largest bubbles observed (300 μ).

In studying the changes that occurred in orange foam with time, LaBelle noted that the foams pictured in figure 185 increased, according to time (age), in light transmittance (I) from 0.11, 0.20, 0.30, to 0.57 per cent. The proportion of incident light transmitted through a foam is inversely related to the number of bubbles or to the interfacial surface (specific surface), which is subsequently related to the mean bubble diameter and bubble density. A progressive increase in light transmittance (I) or orange foams occurs with age or time. This relationship is adequately expressed for wet foams by plotting the square of the transmittance (I^2) against the time (θ), and the results show a linear correlation between the two variables (figure 186). Since the abscissa is a time variable, the slope of the line, 0.00186, represents the rate at which the bubble size is changing. Transmittance data can be used to estimate bubble size in quality control of foams by passing a beam of light through a cell inserted in the line during the drying of citrus juices by foam-mat procedures.

In applying the foam-mat procedure to citrus juices, Berry *et al.* (1965*c*) determined the effects of different drying times of the foams on the final moisture content of the powder (figure 187). These moisture–time curves were determined at 10-degree (Fahrenheit) intervals between 160 and 190° (71 and 88° C.). Grapefruit foams dried for 15 to 26 minutes at 160° F. produced powders with final moisture contents between 3.4 and 2.6 per cent. The curve at 170° F. (77° C.) and 13 to 21 minutes' drying time produced powders with

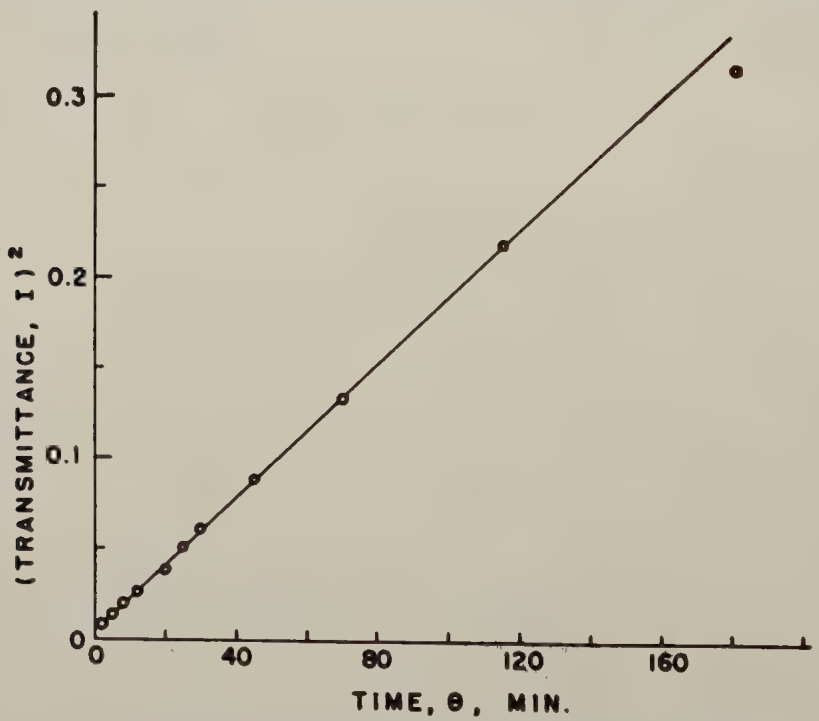

Fig. 186. Square of transmittance and aging in orange foam: $1/K_s = \Delta I^2/\Delta\theta$. (From LaBelle, 1966.)

3.6 to 2.4 per cent moisture. The powders produced at 180° F. (82° C.) and dried between 18 and 26 minutes varied in moisture content from 2.5 to 1.9 per cent. At 190° F. and 8 to 15 minutes' drying time, the moisture content of the products was 2.8 to 1.7 per cent.

Taste tests that were compared to a reference sample indicated slight heat damage and burnt flavor in samples dried at 180° F. for 26 minutes and at 190° F. for 11 and 15 minutes. Berry and his co-workers noted that curves

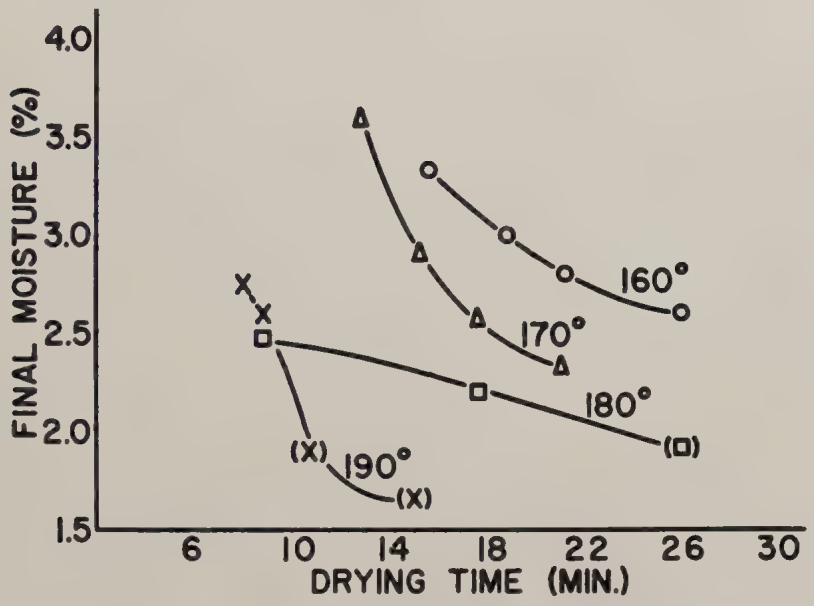

Fig. 187. The relationship of final moisture content of foam-mat dried grapefruit powders to drying time and drying temperature. Symbols in parentheses indicate samples in which a flavor change was detectable at the 0.05 percentage level of significance when compared with a reference sample. (From Berry *et al.*, 1965*c*.)

representing the residual moisture content of orange powders, expressed as a function of the drying time, had steeper slopes than similar curves for grapefruit powders. This relationship indicated that comparatively higher drying temperatures or longer drying periods could be used with grapefruit than with orange concentrate without flavor changes. Grapefruit concentrate could be dried at 170° F. foam temperature without its flavor characteristics being detectably different from the reference sample. Orange foams dried at 170° F. for 14 minutes or more produced powders with detectable flavor changes that were different from the reference sample. Orange powders produced at 180° F. with a drying period of 14 minutes or more had flavor changes that were significantly different from the reference standard. Grapefruit powders dried at this temperature possessed no detectable flavor differences, except in the sample dried for 26 minutes (see figure 187). Orange powders produced at

temperatures above 180° F. had detectable heat damage, but grapefruit foams could be dried at 190° F. for less than 10 minutes without heat damage. It appears that the flavor of grapefruit powders proved to be more stable at higher temperatures and longer drying times than that of orange powders. In these experiments, all the grapefruit concentrates that were dried at 160 and 170° F., and all at 180° F. except those dried for 26 minutes, had total acidity, ascorbic acid, and color measurements within normal amounts.

Storage Data on Foam-Mat Dried Grapefruit Powders. There is a significant relationship between the moisture content and chemical stability of dehydrated food substances. However, this relationship between 2 variables is not a perfect correlation; consequently, other factors are partially involved, such as (1) the reactivity of the chemical components of the dried material; (2) the hydrophilic properties, which include the tenacity with which the water molecules are held by the colloidal or other particles of the material; and (3) finally (but not least), the stability of the dried material under storage conditions at various temperatures.

In the experiments described here, grapefruit concentrates were dried at foam temperatures of 160, 170, 180, and 190° F., and at each temperature,

TABLE 193

RELATIONSHIP BETWEEN DRYING CONDITIONS AND TIME REQUIRED AT 70° F. (21° C.) WITH CaO DESICCANT FOR LOWERING MOISTURE CONTENT TO APPROXIMATELY 1 PER CENT.*

Foam-mat-drying conditions			Original moisture (per cent)	Weeks	Final moisture (per cent)
Temperatures		Time (minutes)			
Degrees F.	Degrees C.				
160	71	23.00	2.80	21	1.08
		20.00	2.88	20	1.12
		17.50	3.45	20	1.29
		15.00	3.70	22	1.15
170	77	20.00	2.50	22	1.16
		17.50	2.62	19	1.19
		15.00	3.05	19	1.24
		13.25	3.82	22	1.28
180	82	17.50	2.61	22	1.22
		11.75	2.48	14	0.90
		8.75	2.48	10	1.03
190	88	13.25	1.65	5	1.25
		10.50	1.70	8	0.97
		8.00	2.42	11	1.08

*From Berry *et al.* (1966*b*).

several different products were made, with drying times from 8 to 23 minutes. To determine the initial quality of each sample before being placed in storage, it was compared with a reference sample prepared under mild conditions (160° F. for 15 minutes).

The data given in the 2 accompanying tables (193 and 194) were recorded by Berry *et al.* (1966*b*). There was an association between the foam-mat drying conditions and the time required at 70° F. (21° C.) for an "in-package" desiccant (CaO) to reduce the moisture content of the powder to approximately 1 per cent (table 193). The desiccation time varied considerably among samples dried at different temperatures by the foam-mat procedure. Grapefruit samples prepared at 160 and 170° F. required, on the average, a 20-week desiccation period; those samples dried at 180 and 190° required periods ranging from 10 to 22 and 5 to 11 weeks, respectively (71, 77, 82, and 88° C.). The time required to reach the desired moisture content (1 to 1.25 per cent) extended from 5 to 22 weeks, depending on the drying temperature. The original moisture contents of the powders (column 4) decreased parallel with the desiccation time in weeks required to reduce the moisture values to the required level.

The relative effects of storage temperatures of 85 and 70° F. (29.4 and 21° C.) on the maintenance of quality of foam-mat dried grapefruit powders were measured by the time in weeks required to detect the earliest organoleptic changes in the stored products. In storage experiments at 85° F., powders prepared at 190° F. had the longest storage lives before the development of detectable organoleptic changes in the reconstituted juice (table 194). The samples dried individually at foam temperatures of 160, 170, 180, and 190° F. had, on the average, corresponding storage periods of 6, 5, 7, and 10 weeks before the earliest organoleptic changes were detected.

These differences are not easily explained. Chemical differences of the powders may have been initiated and accelerated during the foam-drying procedure. At the beginning of the storage periods, all samples had about the same moisture content, which indicates that differences in storage life in these experiments cannot be attributed to the effects of moisture differences.

The data in table 194 also show the relative organoleptic characteristics of foam-mat dried grapefruit powders stored at 70° F. The powders dried at foam-mat temperatures of 160° F. had an average storage life of 36 weeks; at 170°, 33 weeks; at 180°, 32 weeks; and at 190°, almost 30 weeks. The storage data obtained at 70° F. were contradictory to those at 85°, where the samples with longer storage lives were those that had been dried at the higher foam temperature of 190° F.

In order to enhance the flavor of foam-mat dried grapefruit powders, Berry *et al.* (1966*a*) added various amounts of essential oil flavoring constituents to either grapefruit concentrates or powders, and then determined their stability

TABLE 194

RELATIVE STORAGE DURABILITY OF FOAM-MAT-DRIED GRAPEFRUIT POWDERS AT 85 AND 70° F. (29.4 AND 21° C.).*

Foam-mat-drying conditions			Earliest detectable organoleptic change†	
Temperatures		Time (minutes)	Weeks at 85°	Weeks at 70°
Degrees F.	Degrees C.			
160	71	23.00	7	38
		20.00	5	32
		17.50	6	38
		15.00	5	37
170	77	20.00	7	38+
		17.50	5	31
		15.00	4	32
		13.25	4	31
180	82	17.50	7	25+
		11.75	8	35+
		8.75	5	36+
190	88	13.25	9	32
		10.50	12	24
		8.00	10	33

*From Berry *et al.* (1966*b*).
†Significant at the 5 per cent level.

TABLE 195

THICKNESS AND SHEAR STRENGTH OF DISKS FORMED FROM GRAPEFRUIT CRYSTALS OF DIFFERENT MOISTURE CONTENTS AT DIFFERENT APPLIED PRESSURES.*

Disk-forming pressures psi ($\times 10^2$)	Thickness (inches $\times 10^{-3}$)			Shear strength (shear to break disk, pounds)		
	Moisture contents (per cent)			Moisture contents (per cent)		
	0.9	1.3	1.6	0.9	1.3	1.6
38	147	140	143	345	660	355
76	126	125	128	845	930	775
115	119	116	120	1,110	1,325	1,025
134	111	108	113	1,400	2,325	1,590
153	114	109	104	1,225	1,490	1,580
191	114	106	106	965	1,375	1,525

*From Berry *et al.* (1967*b*).
All values are averages of 3 determinations. Thickness, $\pm 3 \times 10^{-3}$ inches; shear, ± 70 pounds.
One square inch equals 6.45 cm^2; one pound equals 0.454 kilogram; one linear inch equals about 2.5 cm.

factors in storage at 85° F. (29.4° C.). As a standard of comparison for flavor equivalent, "locked-in" oil—grapefruit oil encapsulated in crystalline sugar by a patented process—was added to the powders at the 0.005 per cent concentration. The equivalent flavor level of peel oil (cold-pressed grapefruit oil) was 0.0076 per cent; for essence oil, it was 0.078 per cent. The standard sample flavored with locked-in oil had a storage life of 12 weeks at 85° F. before a detectable change in flavor occurred. The samples flavored with cold-pressed peel oil or with essence oil had developed detectable flavor differences after a 6-week storage period.

High-Density Citrus Powders in Relation to Reconstitutive Characteristics. It has been observed that juice reconstituted from grapefruit powders often develops a milky and unnatural appearance, especially if the powders have been produced with certain stabilizing agents. However, the latter may be highly desirable in the foam-mat procedure for the purpose of stabilizing the foam and thus enhancing the liberation of moisture from the interfacial surfaces of the bubbles. The unnatural appearance of the reconstituted juice is caused by the dispersion of microscopic gas bubbles throughout the solution.

Berry *et al.* (1965*b*) reported that when grapefruit powder was subjected to high vacuum for several days to allow the diffusion of the adsorbed gases, there occurred considerable reduction in the dispersed air bubbles, thus reducing to some extent the foam layer of the reconstituted juice. The bubbles in the powder were derived from the foam, and were retained in the powder during the dehydration process. Wagner *et al.* (1964) stated that if the bulk density of the powders could be increased, it would serve to eliminate a portion of the occluded gas, and thus improve the appearance of the reconstituted juice.

Foam-mat dried citrus powders of high densities usually have superior reconstitutive characteristics. Higher densities may be obtained by subjecting the powders to pressure. Berry *et al.* (1967*b*) studied the relationships of 3 variables: (1) pressure applied, (2) moisture of the powder, and (3) the degree of increase in density of foam-mat dried grapefruit powders. Grapefruit crystals with varying moisture contents of 0.9, 1.3, and 1.6 per cent were subjected to pressures ranging from 3,800 to 19,100 pounds per square inch by a ram-driven hydraulic press. These small, firm, compressed disks were subsequently tested in a shear press to determine the amount of shear required for breakage. The shear strength was related to the degree of firmness as well as to the density of the crystals.

The data in table 195 show that the disk thickness decreased with increased pressure except at the higher pressures of 15,300 and 19,100 psi. The crystals with 1.3 per cent moisture produced disks, under 3,800 psi, considerably harder than those produced from crystals containing either the 0.9 or 1.6

per cent moisture contents. The shear required to break these disks was 660 pounds—significantly higher than the 345 and 355 pounds reported for disks produced at the other 2 moisture levels. The pressure data recorded for grapefruit crystals were usually maximum on samples with an optimum moisture content of 1.3 per cent. The thickness of the disks ranged from 0.147 at 0.9 per cent moisture to a low of 0.104 inch for 1.6 per cent moisture. The shear strength of the disks ranged from a low of 345 pounds for 0.9 per cent moisture samples produced at the lowest pressure of 3,800 psi to a high of 2,325 pounds in the 1.3 per cent moisture sample produced under a pressure of 13,400 psi. Berry and his associates concluded that 13,000 to 16,000 psi would be enough pressure to produce disks with maximum shear strength from both orange and grapefruit crystals.

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Subject Index

OUTLINE OF THE VARIOUS TISSUES AND THEIR PRINCIPAL CONSTITUENTS IN RELATION TO THE WHOLE GRAPEFRUIT

The following information should be useful when using the Index:

PEEL (RIND)

FLAVEDO (epicarp, exterior peel, with oil glands). Contains carotenoid pigments, vitamins, and essential oils.

ALBEDO (mesocarp, interior peel). Contains celluloses, soluble carbohydrates, pectin, protopectin, flavonoids, amino acids, vitamins, and minerals.

PULP (endocarp, edible portion)

SEGMENTS covered by segment membranes; JUICE VESICLES (sacs) in the segments; JUICE extracted from the juice vesicles. Pulp contains carbohydrates, glucose, fructose, sucrose, citric acid, vitamin C, vitamin B-complex, minerals, and many other nutrients.

RAG (carpellary membranes, segment walls and tissues between them, and the pulpy central core, or axis.) Contains waste and small amounts of fiber, minerals, sugars, proteins, and pectins used in by-products.

SEEDS

HULLS contain crude fiber and cellulose; KERNELS contain oils and protein.

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